

Corrosion

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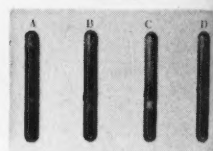
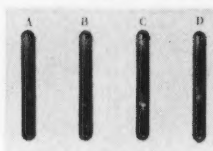
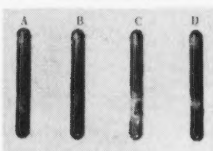
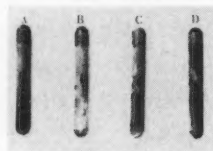
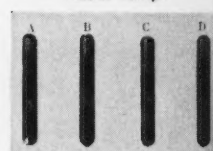
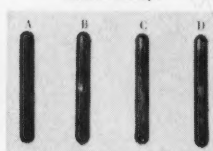
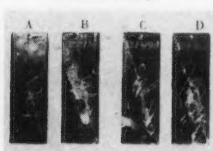
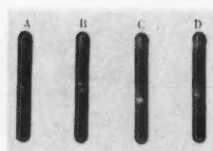
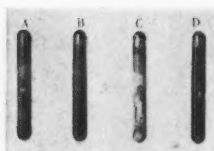
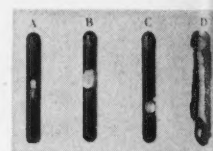
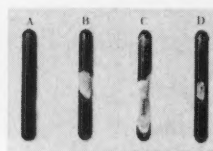
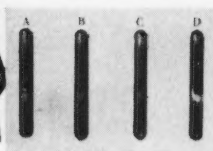
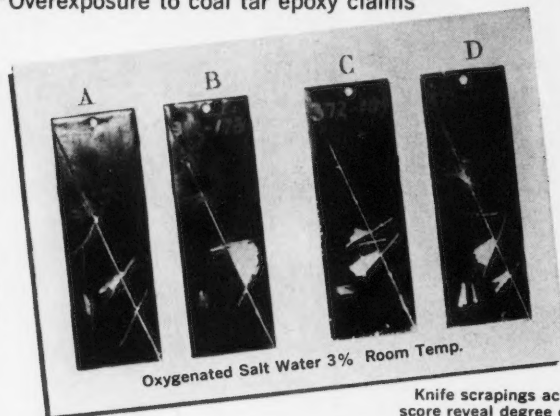


Complete Contents on Pages 2 and 3

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THE TESTS: The rods and panels illustrated here were carefully coated to the four manufacturers' specifications and then subjected for three months to the corrosive agents indicated above. The wide range of tests included varying concentrations of chemicals, ambient and elevated temperatures, weathering and all usual types of exposure.

THE RESULTS: Some results were predictable, such as the failure of all four coatings to withstand aromatic petroleum hydrocarbon solvent. But striking differences occurred in such tests as oxygenated salt water, when only Coating A (Amercoat No. 78) resisted undercutting. Products B, C and D were all blistered and undercut to varying degrees. In none of the remaining tests was Amercoat No. 78 excelled, and in most cases it showed marked superiority.

CONFIRMATION: The properties attributed to No. 78 in these tests have been substantiated repeatedly in actual field use. Applicators like it because it (1) gives dependable, all-around protection, (2) sprays more easily than competitive products, (3) builds thick films without difficulty because of its higher solids content, and (4) dries thoroughly but at a moderate rate, avoiding the extremes of prolonged tackiness and fast-dried brittleness.

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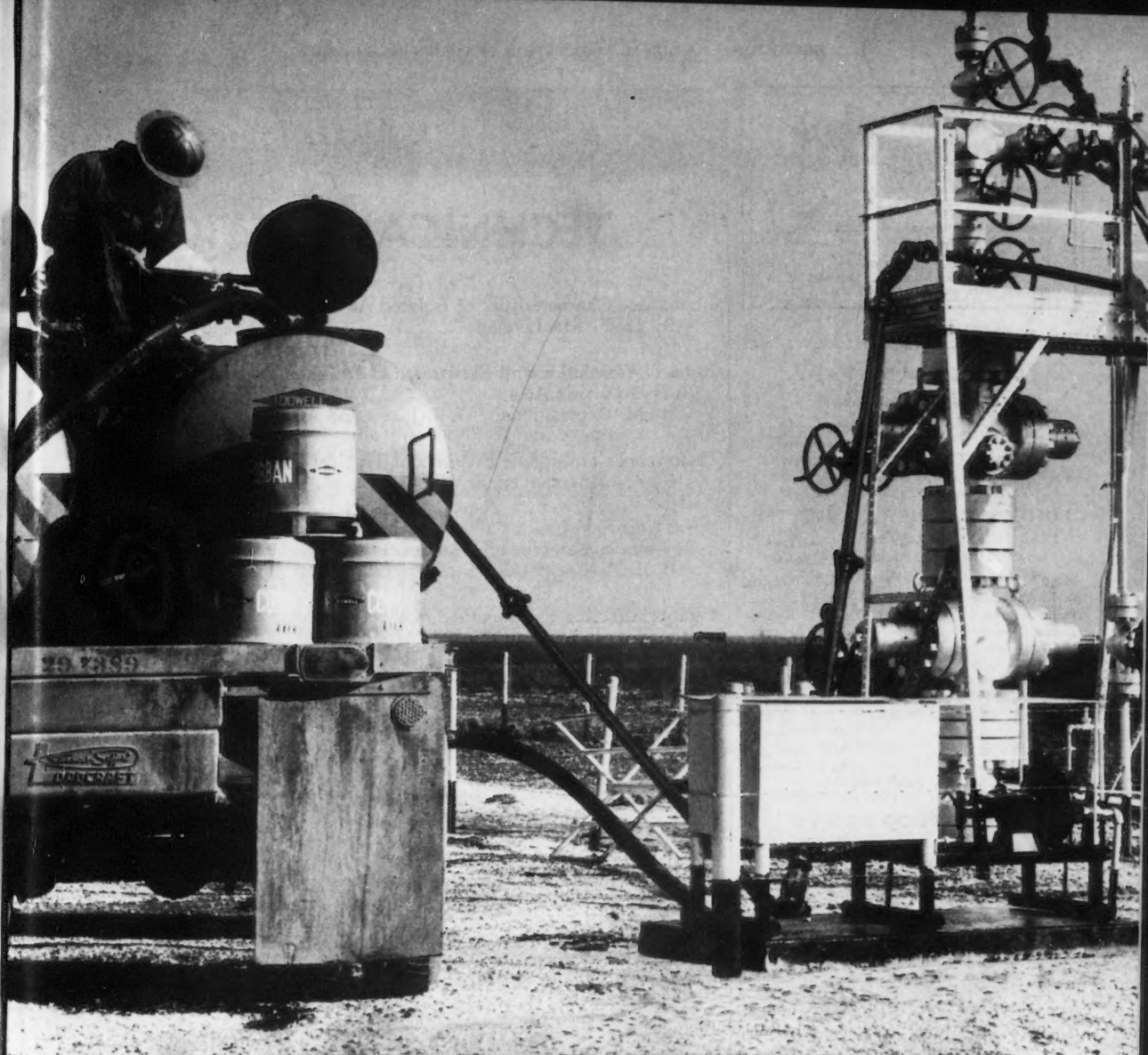
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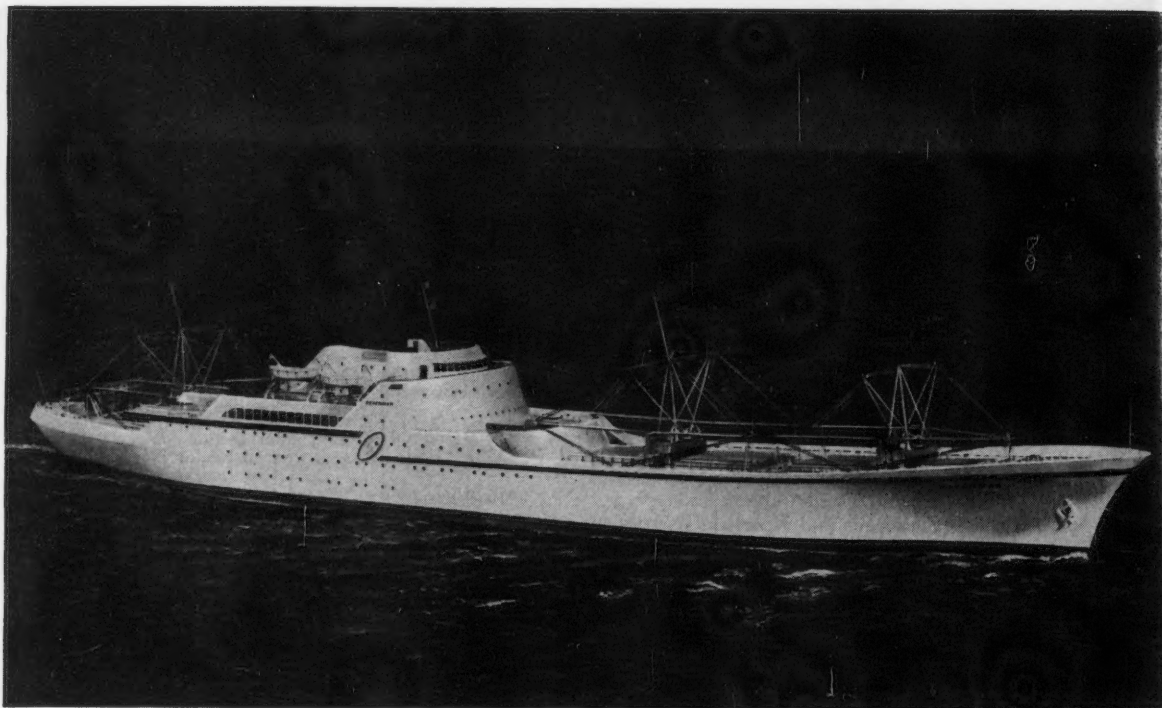
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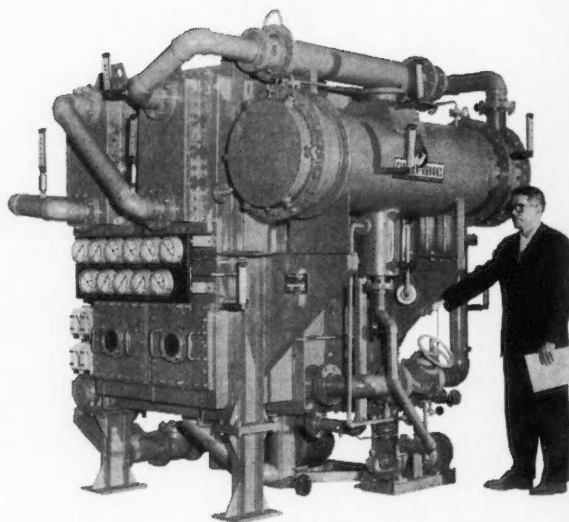
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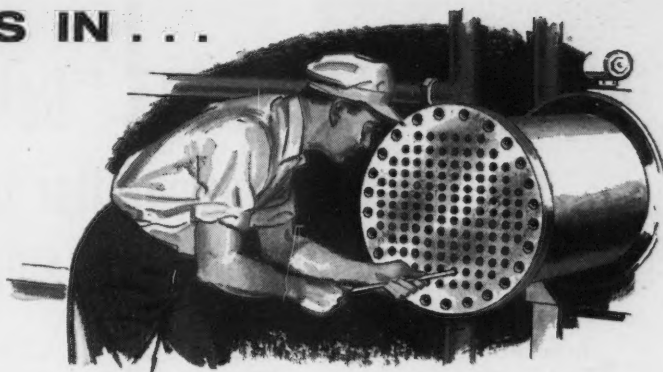
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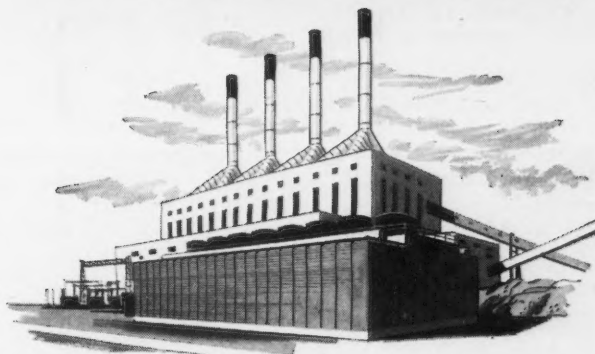
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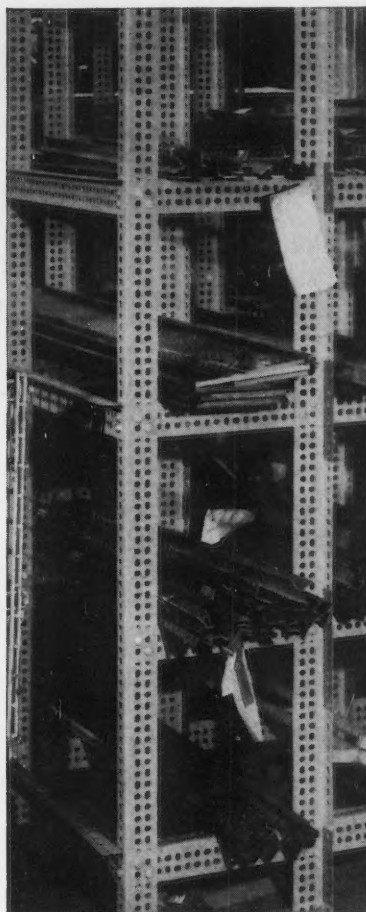


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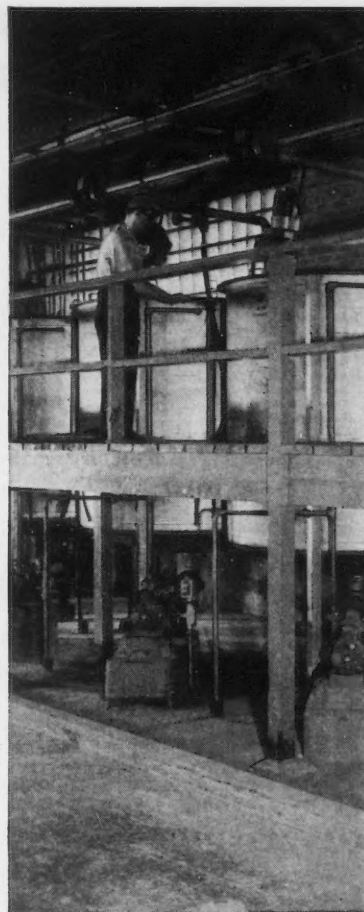
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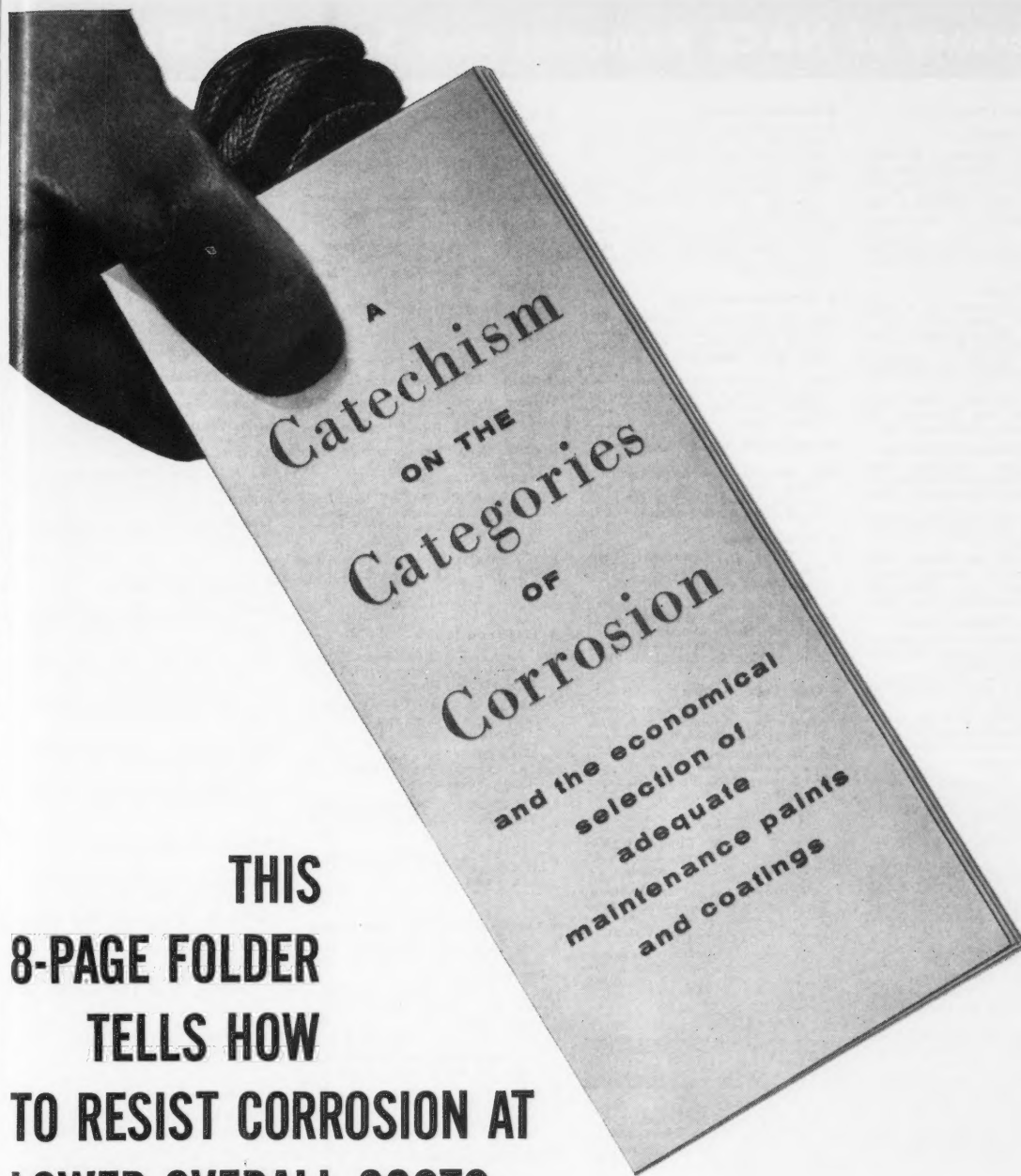
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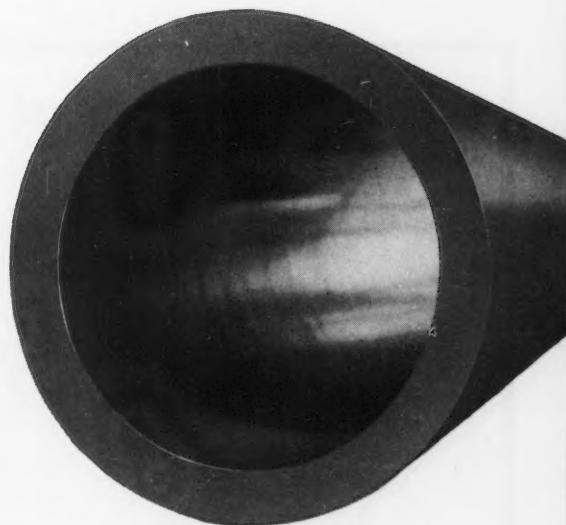
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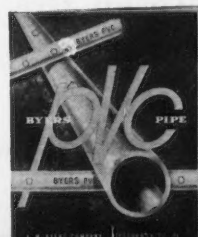
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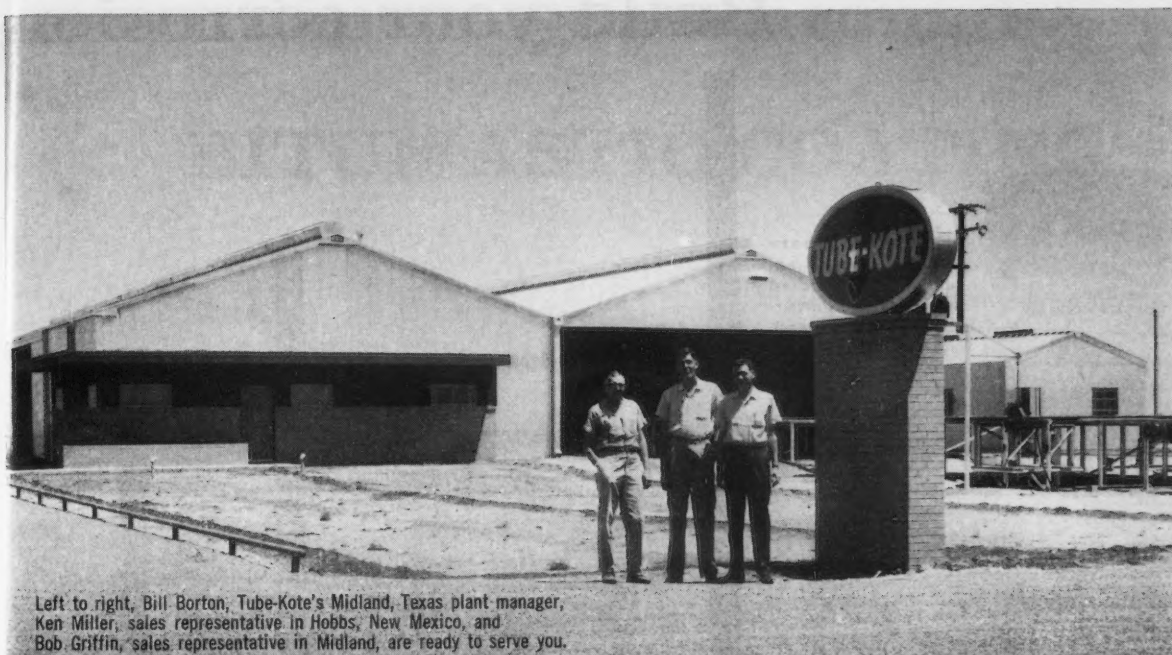
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Impedance Characteristics Of Isolated Aluminum Oxide Films*

By D. F. MacLENNAN

Introduction

WHEN ALUMINUM is made the anode in a cell containing sulfuric acid solution as the electrolyte, a film of aluminum oxide forms on the metal. This film, because of the solvent action of the acid, is porous and may be formed to a considerable thickness. Such films are of interest because they protect the aluminum metal from corrosion. The corrosion protection is greatly increased by treating the film with hot water or steam.¹ The work described here was carried out in order to gain information about these sealing treatments.

Burwell and May² have investigated techniques for quantitatively studying the permeability characteristics of anodic aluminum oxide films. They examined films isolated from the metal by the action of mercuric chloride solutions, and also films prepared by complete anodization of aluminum foil. They found that the films liberated by mercuric chloride solution were less permeable. They also found that continued exposure to the anodizing electrolyte after anodizing resulted in a considerable increase in permeability.

One of the investigations reported by Burwell and May was the measurement of the electrical characteristics of the film immersed in solutions of electrolyte. A similar type of measurement, with modifications, was used in the work described here. The films used in this work were isolated by dissolving the aluminum metal in iodine-methanol solution as recommended by Pryor and Keir.³ This solution gave clean, uncontaminated films.

The treatment of the anodized sample with hot water or steam causes the formation of boehmite.⁴ There are two theories which attempt to explain this. One theory proposes that the boehmite is formed by corrosion of the aluminum metal, while the other contends that it is formed by hydration of the pre-existing aluminum oxide.

About the Author



D. F. MacLENNAN is a member of the Physical Chemistry Section, Naval Research Establishment, Dartmouth, Nova Scotia. He is engaged in a study of the properties of oxide films formed on aluminum and aluminum alloys. He received BSc (1949) and MSc (1951) degrees from Dalhousie University and a PhD (1954) degree from McGill University.

Hart⁵ has investigated the action of boiling distilled water on aluminum, including mechanically polished, electropolished and anodized specimens. Diffraction patterns indicated that boehmite thickness increased with immersion time. Hart concluded that the boehmite was produced by the reaction of hydroxyl ions with aluminum ions which move outward from the metal. Kerr⁶ presented further evidence for this theory. He carried out an electron diffraction study on films, formed in ammonium borate solutions, which were partially isolated from the metal. He found that the boehmite pattern was present on the oxide film near the edges of the metal but absent from the center of large areas of film. He concluded that the boehmite is formed only in the immediate neighborhood of aluminum.

Work with much thicker aluminum oxide films, however, indicates that these are hydrated to boehmite. Spooner,⁷ working with films formed in sulfuric acid solution, has shown by means of weight gain measurements and electron

Abstract

Aluminum oxide films, prepared by anodizing aluminum in sulfuric acid solution, were isolated from the metal and studied. An impedance cell was developed in which the electrical properties of the films were investigated.

Results indicate that the pores in the film are partially blocked by hot water or steam sealing treatments. Also, it is shown that the boehmite, formed during sealing, results from hydration of the pre-existing oxide film rather than from corrosion of the aluminum metal. 3.84

diffraction patterns that water sealing of films is effective on both attached and detached films. This shows that in this case the sealing occurs by hydration of the oxide and that the presence of metallic aluminum is unnecessary.

The present investigation shows that the measured electrical characteristics of isolated thick films are dependent upon the sealing treatment but are not influenced by the presence or absence of aluminum during the sealing operation.

Experimental

Preparation of Films.

Anodizing. Strips of 2S (99.0 percent +) aluminum sheet, three inches by one inch by 0.02 inch, were cleaned by wiping with varsol. They were partially coated with electroplating tape so that a well defined area of one square inch on one side of each sample was exposed. Each aluminum strip was then made the anode in a 15 percent (by volume) sulfuric acid solution, and a current of 50 milliamperes was passed for 15 minutes. The sample was then thoroughly rinsed with distilled water and allowed to dry.

Isolating. The edges of the unanodized side of the dry sample were masked off with polystyrene cement in order to protect them from the isolating solution and thus provide a metal frame for the isolated film. The sample was then placed in an iodine-methanol solution (10 gm iodine/100 ml methanol). This solution dissolved the aluminum from the unanodized side and left the oxide film unattacked. After isolation, the film was given several rinses in methanol in

* Submitted for publication June 27, 1958.

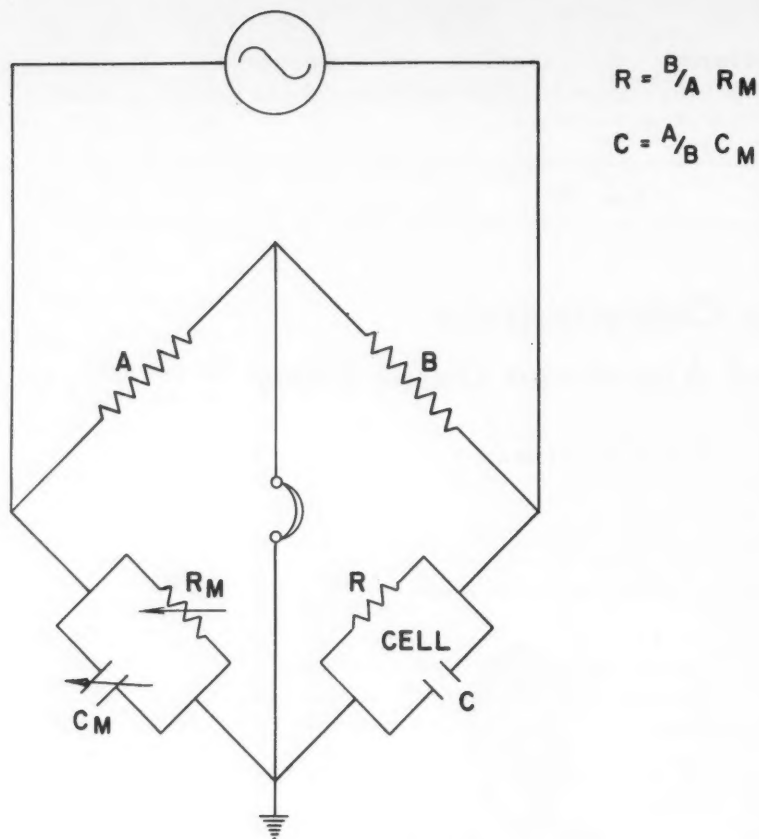


Figure 1—Circuit for impedance measurements.

order to remove traces of the solution. The oxide film obtained was transparent, approximately six microns thick (measured by micrometer) and quite brittle.

Sealing. The sealing operation was carried out either before or after the isolation of the film. The steam treated films were suspended in steam at atmospheric pressure for 15 minutes. The water treated films were suspended in boiling distilled water for 15 minutes. This water was prepared by boiling for 15 minutes in order to expel dissolved air, and then cooling under vacuum. The samples were immersed in the cool water, which was then boiled for 15 minutes. It was necessary to cool the water before introducing the samples because isolated films broke when placed directly into hot water.

Types. Five types of films were prepared:

1. Untreated.
2. Fifteen minutes in steam (atmospheric pressure) before isolation.
3. Fifteen minutes in steam (atmospheric pressure) after isolation.
4. Fifteen minutes in boiling water before isolation.
5. Fifteen minutes in boiling water after isolation.

Measurement of Electrical Characteristics.

The impedance cell was made of plastic and consisted of two compartments, each of which was a two-inch cubic box. In one face of each cube, there was a 1/4-inch diameter hole. The compartments were placed side by side, with the holes meeting, so that the two sections were joined through the openings. The cubes were held in this position by elastic bands. Each compartment contained a platinum electrode, placed near the hole.

Impedance measurements were made with an impedance bridge. A decade condenser was connected across the bridge, as shown in Figure 1, in order to balance capacitance. All measurements were made at 1000 cycles.

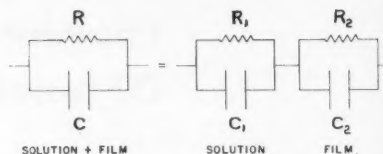
A cell was filled with electrolyte and the impedance measured (R , C). The cell was then emptied, dismantled and reassembled with an aluminum oxide film separating the two compartments. It was refilled with the same electrolyte and the impedance measured again (R , C). Readings were taken at various time intervals during the first two hours that the electrolyte was in contact with the film.

The advantage of this type of cell is its simplicity. It is easily assembled and dismantled. The film fits between the compartments, covering the holes. It is held in position by the mutual pressure

of the compartments and a tight seal is obtained with lightly greased paper gaskets. Thus, it is not necessary to use any cement on the film.

Results

In order to obtain values which are representative of the film, it is necessary to eliminate the solution resistance from the measured quantities. The cell is represented as two parallel resistance-capacitance circuits in series:



The values of R_2 and C_2 are calculated by subtracting the circuit R_1 and C_1 from the circuit R and C . Since C_1 in the present work was always zero, the equations are simplified to:

$$R_2 = \frac{(R - R_1)^2 + (RR_1\omega C)^2}{(R - R_1) - R_1(R\omega C)^2} \quad (1)$$

and

$$C_2 = \frac{R^2 C}{(R - R_1)^2 + (RR_1\omega C)^2} \quad (2)$$

where ω is 2π times the frequency of the alternating current.

The film may be represented by a point on a graph in which R_2 is plotted against C_2 . In Figures 2 and 3, the results are presented in this way. For each film two points, joined by a straight line, are given. These points represent the film at different times. In general, the point with the higher resistance was obtained during the first 10 minutes that the solution was in contact with the film. The other point was obtained between one and two hours later. The changes which occur in resistance and capacitance during this period are small and permit direct comparison of the various films. Results of at least two similar films are given for each set of conditions. In Figure 3, results are plotted on logarithmic paper in order to facilitate comparisons.

Results show that there is a certain amount of variation in the characteristics of films which were prepared in the same way. This is due to the fact that each film was individually prepared at room temperature and slight temperature variations during anodizing would result in film differences. However, these differences are very small compared to the changes observed in films that were given different treatments, and thus do not interfere with the interpretation of results.

Discussion

The widely accepted structure for this type of film³ is a system of parallel pores uniformly distributed over the entire surface, approximately perpendicular to the surface, and extending through almost the entire oxide layer. A thin imperme-

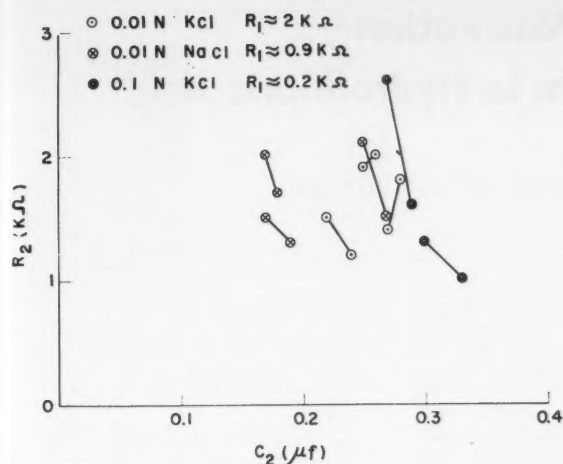


Figure 2—Impedance characteristics of untreated films in various electrolytes.

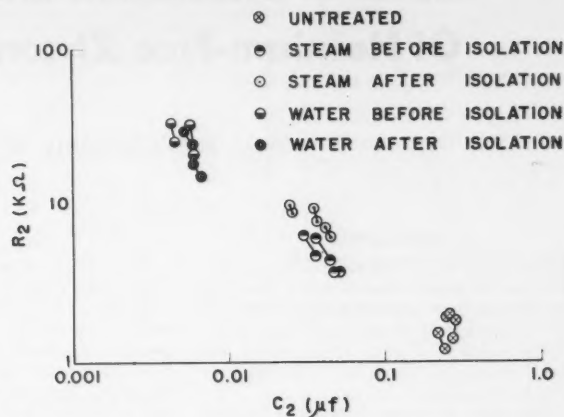


Figure 3—Impedance characteristics, in 0.01N KCl, of films after various treatments.

able layer of oxide exists between the bases of the pores and the metal. The results presented here are compatible with this structure.

The impedance characteristics of untreated aluminum oxide films in various electrolytes, plotted in Figure 2, show that these quantities do not vary with the electrolyte resistance. If the impermeable layer of oxide had been destroyed or damaged in the isolating process, the resistance exhibited by the remaining porous film would be dependent upon the resistance of the solution carrying current through the pores. However, when solution resistance was varied by a factor of ten, the variations of the film parameters were small and random and can be attributed to differences in the individual films. This indicates that the measured quantities are characteristics of the impermeable portion of the film, and thus shows that the iodine-methanol solution isolates the complete film.

Impedance characteristics of films, after various treatments, are plotted in Figure 3. The resistances of steam treated films are significantly higher and the capacitances significantly lower than those of untreated ones. These changes are due to a decrease in the size of the pores. Since films treated after isolation from the metal exhibit the same characteristics as those treated while attached, it appears that the boehmite must be formed by hydration of the oxide film. The hydrated form of the oxide occupies a larger volume than the original oxide and, therefore, hydration will decrease the pore size.

The resistances of hot water treated films are significantly higher and the capacitances significantly lower than those of the steam treated ones. This means that in a given period of time, more hydration occurs in hot water than in steam at atmospheric pressure. A larger volume of boehmite is formed and the pores are blocked to a greater extent. It is noted that in this case, also, the impedance characteristics are not affected by the presence or absence of aluminum metal during sealing.

Hunter and Fowle⁹ have measured barrier layer thicknesses by measuring the voltages required to pass a direct current through films on aluminum anodes. They reported that the barrier layer thickness was reduced by the sealing process. However, the impedance results reported here show that the sealing process increases the resistance of the film to the passage of an alternating current between two electrolyte solutions.

A possible explanation of this discrepancy is that during the sealing process, part of the barrier layer is converted to boehmite. The boehmite offers negligible resistance to the current in the Hunter and Fowle type of measurement and, therefore, the thickness of the unconverted barrier layer is measured. However, the boehmite offers considerable resistance to the current in the impedance type of measurement and, therefore, the results obtained depend on the barrier layer and boehmite. Further work is required to clarify this point.

Conclusions

1. The film formed by anodizing aluminum in sulfuric acid, which consists of a thick porous outer layer and a thin impermeable inner layer, can be removed from the metal by the iodine-methanol method without damaging the thin layer.
2. The sealing treatment causes a partial blockage of the film pores.
3. Boehmite is formed by hydration of the pre-existing oxide film.
4. In a given period of time, more hydration occurs in hot water than in steam at atmospheric pressure.

Acknowledgment

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Any discussion of this article not published above
will appear in the December, 1959 issue

Rates of Dissolution and Passivation Of Hafnium-Free Zirconium in Hydrofluoric Acid*

By M. E. STRAUMANIS, W. J. JAMES and A. S. NEIMAN

Introduction

EXCELLENT CORROSION resistance to most mineral acids is one of the outstanding non-nuclear properties of Zr.^{1,2} Concentrated sulfuric and phosphoric acids and aqua regia attack the metal slowly although hydrofluoric acid attacks it quickly.³

The older literature is given in Mellor⁴ and in the "Corrosion Handbook".⁵ Fontana⁶ and Miller⁷ stated that Zr was rapidly attacked by HF at all concentrations. In general, it was found that Hf-free Zr (containing less than 0.1 percent Hf) was more corrosion resistant than the metal containing the usual 2.5 percent Hf.¹ Johnson and Hill⁸ observed that such a Zr was not attacked by HF in 2 day tests at concentrations of 0.005N and less. At concentrations from 0.02 to 0.04N the formation of a gray film on the surface of the dissolving metal was reported. This film turned black during the reaction at acid concentrations of 0.05 to 0.1N.

Quantitative studies on rates of dissolution of Zr in HF were made by Baumrucker⁹ and continued by Smith and Hill,⁹ who employed radioactive Zr⁹⁰ as a tracer to study the rates of dissolution. The two latter authors found that the plot, rate of dissolution versus concentration of HF (up to 0.5N), is essentially a straight line, independent of the oxygen concentration in the gas above the acid, and independent of small additions of NO₃⁻, Cl⁻, ClO₄⁻, F⁻, HF₂⁻ and K⁺ to the acid.

From this survey it can be seen that there has been very little quantitative investigation of the dissolution of Zr in HF. The intention of the present study therefore is (1) to confirm the straight line relationship found by Smith and Hill⁹ using an entirely different method of rate measurement, (2) to investigate the influence on rate by additions of some salts to the acid, (3) to find the composition of surface layers formed on dissolving Zr and, (4) to follow the potential changes of this metal during various stages of corrosion and passivation.

Materials, Equipment and Procedure

The low-Hf Zr⁽¹⁾ contained in percent by weight: O₂—0.11, N₂—0.005, Fe—0.04, Hf—0.01.

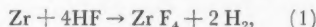
After the metal was rolled, the stress was relieved by annealing in vacuum for 30 minutes at 700 C. From this sheet, approximately 1.6 mm thick, the samples for the experiments were cut.

* Submitted for publication August 11, 1958. This paper was extracted from a thesis by Mr. Neiman submitted in partial fulfillment of the requirement for the master's degree to the Graduate School of the University of Missouri.

(1) Obtained from U. S. Bureau of Mines, Albany, Oregon.

Hydrofluoric acid pro analyse (48 percent) was used for the preparation of solutions of various normalities. The normalities were determined by titration against standardized CO₂-free NaOH, in the presence of phenolphthalein indicator. All the operations with F⁻ containing solutions were performed in polyethylene, polystyrene, or wax lined glass vessels.

The rate of dissolution was followed by measuring the rate of hydrogen evolution. As the reaction of Zr with HF proceeds according to equation (1)³



one gm of Zr dissolved should correspond to 491.44 cm³ of hydrogen at standard conditions. The validity of reaction (1) and also the purity of Zr used, was checked with an apparatus previously described^{9,10} (see Table 1).

This average deviation of ± 0.2 percent from the theoretical value shows that the Zr is of high purity and that Equation (1) is correct. The equipment for the determination of rates was essentially the same as used previously in similar studies with Ti.¹¹

The Zr sample was mounted on an ebonite foot 5 mm from the center of the stirring rod at an angle of 45 degrees toward the direction of rotation. A stirring speed of 200 rpm was maintained throughout the experiments. The volume of the acid was 125 ml, and the temperature in all experiments was 25 ± 0.1 C.

The Zr samples were cut from the sheet mentioned, finished to the size of one cm², and were mounted in Bakelite.

The rate of dissolution V was calculated from

$$V = \frac{\Delta v}{\Delta t} \times 10^3 \text{ in mm}^3 \text{ cm}^{-2} \text{ min}^{-1} \quad (2)$$

where Δv is the increase in volume (reduced to standard conditions) during the time increment Δt . To convert the mm³ into mg, the factor 2.035/1000 mg should be used.

Abstract

The dissolution reaction of Zr in HF is: $\text{Zr} + 4\text{H}^+ \rightarrow \text{Zr}^{4+} + 2\text{H}_2$. The overall rate of dissolution V at 25 C and under the conditions of the experiments is much faster than that of Ti, and is a linear function of the HF concentration (up to $N = 0.5$): $V = 2000 \text{ N mm}^3 \text{ cm}^{-2} \text{ min}^{-1}$. A black hydride film is formed on Zr during its dissolution.

Fluoride additions (NH₄F, KF, NaF) to the acid initially increase the rate of dissolution. When added in greater amounts, passivation of Zr is caused which is quite complete for NaF additions. The order of increasing effectiveness is $\text{NH}_4\text{F} \rightarrow \text{KF} \rightarrow \text{NaF}$, apparently caused by the decreasing solubility of fluozirconate salt film formed on the metal.

Potential measurements of Zr in HF showed an approach to a steady potential of about -0.74 volt with increasing concentration of the acid. The potential time curves had in general a logarithmic shape, the potential becoming more positive with elapsing time. Fluoride additions caused a breakdown of the initial film, formed during dissolution in pure HF, and a sudden drop of potential down to -1.112 volt (in 6.5 M NH₄F). The potential gradually became more positive, because of the salt film formation on the surface of Zr, until a steady potential was reached. Of the three additions, NaF produced the most positive potential (-0.528 volt) and a complete passivation of Zr. 6.3.20

Rates in Pure HF

The rate of dissolution reached a maximum in 10 to 30 minutes after the start of a run and then decreased quickly, in agreement with the work of Smith and Hill.⁹ A tenacious black film was formed on all specimens except on those in 0.01N acid, where the film seemed to be extremely thin, intermittent and gray in color.

An average of the rates over a range where they were highest and nearly constant was used as a medium maximum rate for each run. Two and more runs were averaged for each concentration. The plot of rate against concentration is shown in Figure 1. It can be seen that there is an essentially straight line rate-concentration relationship with a slight bend at 0.25N HF. The equation for the straight line is about

$$V = 2000 \text{ N mm}^3 \text{ cm}^{-2} \text{ min}^{-1} \quad (3)$$

which means that the rate of dissolution of Zr is much faster than that of Ti¹¹ in the same acid.

TABLE 1—H₂ Evolution From Hf-free Zr in HF in Presence of Hydrogen and Air*

Atmosphere	Zr in gm	Vol of H ₂ in cm ³	Vol calc eq. 1	%
H ₂	0.0624	30.39	30.67	+0.67
	0.0410	20.48	20.15	+1.64
	0.0735	36.32	36.12	+0.55
	0.0639	31.65	31.90	-0.54
	0.0524	25.92	25.75	-0.91
Air	0.0464	22.79	22.80	+0.04
	0.0436	21.40	21.40	± 0.00
	0.0493	24.85	24.20	(+2.68)
Average, last exp. excluded				+0.2%

* Volumes at standard conditions.



MARTIN E. STRAUMANIS, at present research professor of metallurgy at the University of Missouri, School of Mines and Metallurgy, Rolla, holds degrees of Engineer Chemist (1925) and Dr. Chem. (1927) from University of Latvia, Riga. He spent his post-doctorate year at the University of Göttingen working with G. Tamman (metallurgy) as a Fellow of the Rockefeller Foundation. Between 1934 and 1944

he was director of the analytical and X-ray laboratories at the University of Latvia. When forced to leave Latvia in 1944 he devoted three years to research at Institute of Metal Chemistry, University of Marburg, Germany. He came to the United States in December 1947 and is now an American citizen. He is author of 164 articles on metal corrosion, inorganic chemistry, crystal growth and X-ray crystallography.

WILLIAM J. JAMES is research associate professor and associate professor of chemistry in the department of chemical engineering of the University of Missouri School of Mines and Metallurgy. He received his BSc in chemistry from Tufts University and a PhD. in chemistry from Iowa State College. His major work is in the field of X-ray crystallography and physical chemistry.



ALFRED S. NEIMAN is a metallurgist at Stanford Research Institute, Menlo Park, California. His principal field of research is metal brazing and secondly high temperature missile materials. He received a BS and MS in metallurgical engineering in 1953 and 1957 respectively from the Missouri School of Mines.



Rates in HF in Presence of Acid and Salt Additions (Passivation Phenomena)

Nitric Acid Additions, Qualitative Tests

Johnson and Hill¹ found that additions of nitric acid decreased the rate of reaction of Zr with HF.

Four wet samples of Zr with a black film on them were placed in 16, 8, 4, and 2N HNO₃ solutions respectively. No gas evolution was observed. Then HF was added. Gas evolution started with simultaneous breakdown of the black film as soon as the solutions became 0.1N with respect to HF. However, the gas evolution soon decreased with the phenomena occurring most rapidly in the 16N HNO₃. The more dilute the nitric acid, the longer was the time required for passivation. The formation of a very shiny surface² accompanied passivation. The passive specimens then were placed in pure 0.1N HF; the shiny surface changed very quickly to a black film and gas evolution started. However, it was impossible to reproduce the rates of

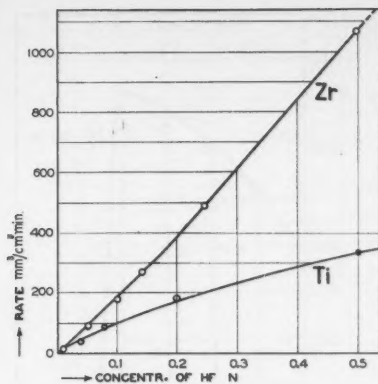


Figure 1—Rate of dissolution of Hf-free Zr and Ti in HF of various concentrations at 25 C. One mm³ corresponds to 0.002035 mg Zr. Each point represents an average of multiple readings.

gas evolution created in such experiments.

Passivation by Fluoride Additions

Additions of alkali fluorides to HF may produce fluorozirconate salt layers¹² on the Zr, causing its passivation, as in the case of Ti.¹³ The same equipment for rate determinations was also utilized for investigation of fluoride additions. At first each sample was reacted for the determination of rate for 100 minutes in pure 0.1N HF. Then, as the most efficient method proved to be the replacement of the entire reaction volume by a newly premixed solution of the desired composition, the solution was poured out from the flask and replaced by a premixed 25 C solution. The time needed to replace the solutions was about three minutes and all runs were continued approximately 100 minutes in this solution. The latter was 0.1N with respect to HF and contained certain amounts of NH₄F, KF or NaF. In Figures 2, 3 and 4 the rates plotted are averages over a time of about 60 minutes after the immersion of the Zr sample into the HF-fluoride mixture. All points on the plots are a mean of at least two runs with a few exceptions, which were single runs. The zero fluoride concentration point on all plots was found by averaging 10 rates of dissolution of Zr at 25 C in 0.1N HF at the 100th minute.

The common feature for all three series of experiments is that the rate of dissolution increased with small additions of fluorides to the acid, then the rate of dissolution decreased abruptly as soon as a certain concentration of fluoride in the acid was reached. The concentrations of fluorides causing passivation were not equal: 3M for NH₄F, 0.5M for KF and only 0.1M for NaF. In all these cases the rate of dissolution dropped severely, but a complete passivation was attained only with NaF additions. The minimum rates achieved by additions of NH₄F, KF and NaF were 80, 20 and 0 mm³/cm² min respectively. In the first two cases the minimum rates were observed 110 minutes after the addition, but in the last one after 50 minutes. In general, reproducibility of the rates was quite good, except for the time-rate curves.

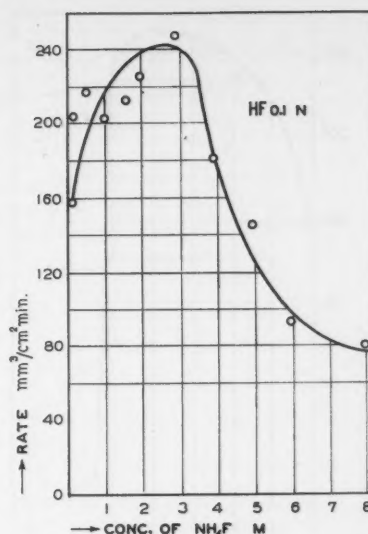


Figure 2—The effect of NH₄F additions (in M) on the rate of dissolution of Zr in 0.1N HF at 25 C.

Passivating Films on Zr

Black Film Formed in HF.

The black film formed on Zr during the dissolution in HF could be removed in a saturated NH₄F solution. Evidently the tenacious black film was undermined and loosened by the passivating film formed in the NH₄F solution. Figure 5 displays the breakdown and replacement of the original black film by the other passivating salt film after treating the sample about 110 minutes in 0.1N HF solution containing various concentrations of NH₄F (in moles/liter). Specimens a and b show flow lines in the black film. These originated from the breakdown of the film exposing an underlying shiny, metallic surface. On the specimen c an iridescent film was observed with small amounts of black film remaining. On the last specimen a more complete bright film had developed. All the films shown in Figure 5 were very tenacious.

The black film, after being removed from the Zr plate by a saturated NH₄F solution, was washed, dried and crushed to powder. X-ray powder diffraction patterns were made of this substance. The pattern showed similarity to the Zr-powder pattern, but with lines on it which definitely belonged to ZrH₂. The formation of a zirconium hydride film is quite possible because of the analogy to the hydrides of Ti,¹⁴ which have been found on the surface of the corroding metal by Ogawa and Watanabe¹⁵ and by Rüdiger, Fischer and Knorr.¹⁶ According to Sidhu, hafnium, a metal very similar to Zr, readily forms hydrides.¹⁷ The fact that Zr also gives hydrides at high temperatures^{18,19} contributes to the possibility of hydride formation at room temperature under conditions of corrosion. The dissolution of the metal occurs through the pores of the black hydride film.

The film is resistive to the action of HF and dissolves completely only at concentrations above ~3N. As already mentioned, HNO₃ destroys the hydride film,

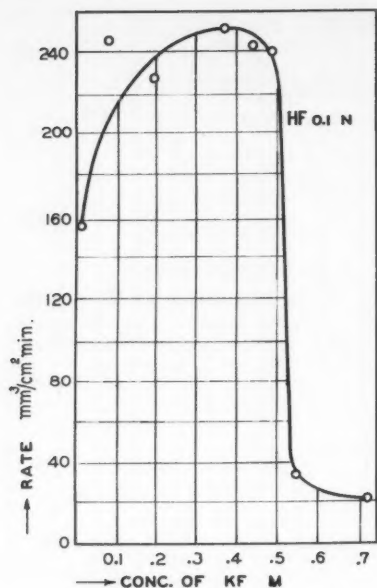
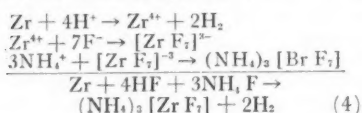


Figure 3—The effect of KF additions in tenths of M on the rate of dissolution of Zr in 0.1N HF at 25 C.

probably replacing it by an invisible oxide film.

The Salt Films.

The films formed in presence of alkali fluorides were salt films. Although no x-ray diffraction patterns could yet be obtained from specimens passivated in NH_4F solutions, the analogy with the films produced in KF solutions, for which the patterns could be obtained, points toward the formation of thin salt films in NH_4F solutions (Equation 4):



The possibility that the salt film contains the hepta fluo zirconate and not the hexa salt is supported by an investigation of Hevesy, Christiansen and Berglund,²⁰ because passivation of Zr occurred in presence of large amounts of NH_4F .

Upon passivation of Zr at KF concentrations over 0.5M a gray salt like film was present on the exposed surfaces of the specimens. With the salt film in place the specimen was mounted on a Philips X-ray diffractometer supplied with a Geiger-Müller counter and a recorder. The pattern obtained was compared with the ASTM tables, for which purpose the "d" values of the lines were calculated. The comparison of these values with those of ASTM cards for certain compounds is given in Table 2.

Table 2 shows that the passivating salt film consists of K_2ZrF_6 and $\text{KF} \cdot 2\text{H}_2\text{O}$. The Zr lines obtained from the substrate could be easily eliminated by comparison with an x-ray picture of pure Zr.

Patterns from the salt film on the specimen passivated in NaF were obtained by both surface and powder camera techniques. However, when compared with

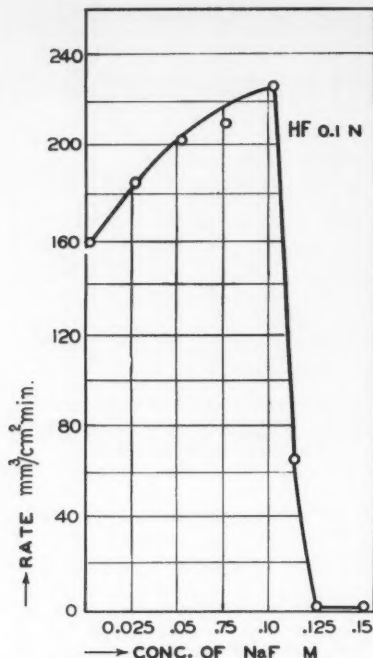


Figure 4—The effect of NaF additions (in hundredths of M) on the rate of dissolution of Zr in 0.1N HF at 25 C.

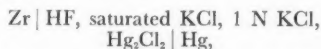
the ASTM tables no respective compound could be found, and the lines of Na_2ZrF_6 were not listed there.

Potentials of Zr in HF

Experimental Arrangement.

Changes in potential of dissolving metals are frequently caused by a formation and breakdown of protective coatings.²¹ Hence, the replacement of old coatings by new ones, as was observed in experiments with Zr, should also be indicated by changes in potential of the metal.

The overall experimental arrangement provided a measurement of the emf of the cell



by means of a potentiometer. The potential of the Zr/acid electrode was then reduced to the hydrogen scale by adding 0.280 volts. The junction potential was eliminated by a salt bridge filled with a concentrated KCl-solution.

The area of 1 cm² of the Zr electrode exposed to the acid was entirely submerged in the solution.

The tube connecting the salt bridge and the acid was filled with 0.14N HF, the lowest concentration for sufficient conductivity to provide adequate sensitivity of the potentiometer.

Before fluoride additions were made the Zr electrode was dissolved with stirring in a 0.1N HF solution for 75 minutes. Then either a solid fluoride was added or the 0.1N HF was replaced by a solution having the desired fluoride and HF concentration. Potential measurements after the addition were continued for 24 hours or longer.

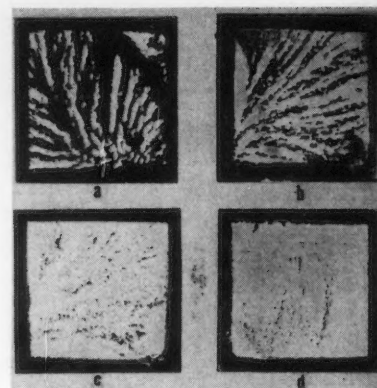


Figure 5—Zr samples with black film on them, treated about 100 minutes in 0.1N HF— NH_4F solutions:
a—3M
b—4M
c—6M
d—8M NH_4F
The samples shown are in Bakelite mountings.

Potentials in Pure HF.

A set of potential-time curves for various concentrations of HF is given in Figure 6. The general trend is that the potential of Zr just after the immersion into the acid quickly becomes more positive with only a gradual increase in potential later, and that the potential decreases (turns more negative) with increasing concentration of the acid (Figure 7). The irregularity of the curves, especially in diluted HF, may be caused by the dissolution of the initial oxide film present on the metal and by the formation of the new hydride film. The approach to a steady potential with time is, therefore, due to an increased rate of dissolution and due to the hydride film formation.

The plot Figure 7 shows that the potential of the Zr electrode with increasing concentration becomes more negative approaching a limiting potential of about -0.740 volt. This behavior is normal and is also observed with other metals (Zn).²²

Potential, and Fluoride Additions.

The effects of fluoride additions on the potential-time curves were very similar in case of NH_4F and KF additions and slightly different when NaF was added. The curves are shown in a single diagram, Figure 8. Thus, it was possible to follow through potential-time curves the breakdown of the black hydride film and the subsequent built-up of the passivating film.

The 6.5M NH_4F (and 0.1N with respect to HF) caused a sharp decrease in potential (at the 75th minute, when the salt was added) indicating the start of the hydride film breakdown and shift of equilibrium to undissociated HF. Simultaneously an increase in the rate of H_2 evolution from the Zr was noticed, decreasing steadily so that at the 515th minute only localized gas evolution occurred at the edges. The NH_4F caused also a sharp decrease in potential down to -1.112 volt, followed by a quick increase up to -1.076 volt. Apparently the breakdown

of the hydride film at this point was finished (visually evident) and the build-up of the salt film started. The growth of the latter showed up in a gradual increase in potential (and decrease in rate of dissolution) reaching a steady potential of -1.015 volt in about 1340 minutes. The $3M$ NH_4F produced passivation to a lesser extent, so that the H_2 evolution did not noticeably decrease even in 1080 minutes, being then greater by far than in $6.5M$ NH_4F at the same time. This result is in accordance with the rate measurements previously described.

Essentially the same curves were obtained using KF as a passivating agent. However, a $0.71M$ fluoride solution was sufficient to produce the same effects as did the $6.5M$ NH_4F solution. A second drop in potential after the KF addition (not observed in NH_4F) was reproducible and even more intense in the case of $1.37M$ KF . From this second minimum the potential gradually became more positive until after 420 minutes a steady potential of -1.030 volt was reached.

The potential-time curve in $0.15M$ NaF differed from the two curves in so far that the potential minimum was only -0.988 volt (Figure 8), and that it increased much faster, finally reaching a far more positive steady potential of -0.528 volt. This more positive potential can account for the very complete passivation of Zr : 7 minutes after the small NaF addition, the H_2 evolution from the sample ceased. The reproducibility of all the potential-time curves was good.

Discussion

Comparison With Previous Studies.

This investigation is not entirely in agreement with the two quantitative studies previously made by other authors.

The formation of a black tenacious film was always observed when Zr dissolved in low concentrations of HF . However, Baumrucker describes the film as being a loose black smut.⁸ As he used a much more concentrated acid ($2.5N$), the high rate of hydrogen evolution and the acid itself may loosen the adherence of the black film to Zr .

Smith and Hill stated that K^+ , F^- and NO_3^- (among others) had no effect on the reaction rate.⁹ However, Figure 3 shows the opposite; hence the concentrations of KF used by the two authors were too low. The presence of HNO_3 also affected the rate, although it was difficult to measure the effect quantitatively.

The Passivation Mechanism.

Two steps in passivation should be distinguished: the one produced by the hydride film and the one by salt additions. It is best for this purpose to analyse the curve (Figure 8). As already mentioned, the oxide film on the surface of Zr may cause a fairly positive potential of the metal in HF . Evidently this film dissolves so fast in HF that it is not possible to observe the drop in potential, but only the rise which is due to formation of a partially protective porous hydride film on the surface of Zr due to hydrogen liberation according to reaction (1).

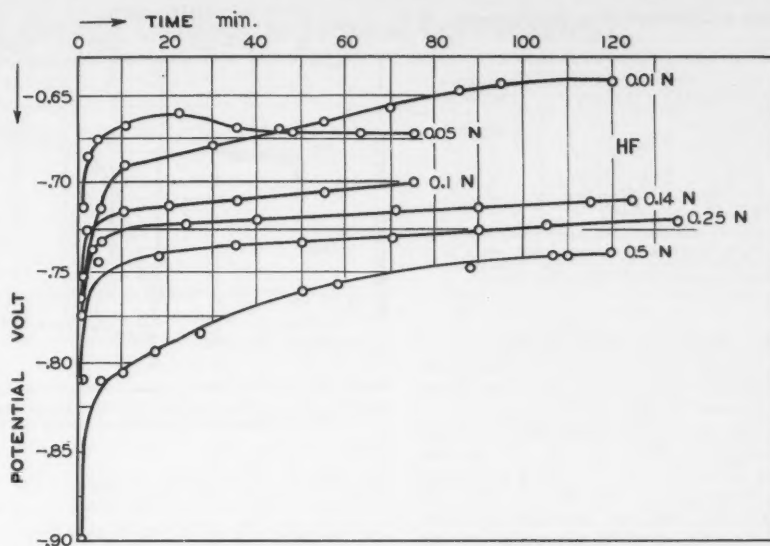


Figure 6—Potential-time curves of Zr in various concentrations of HF .

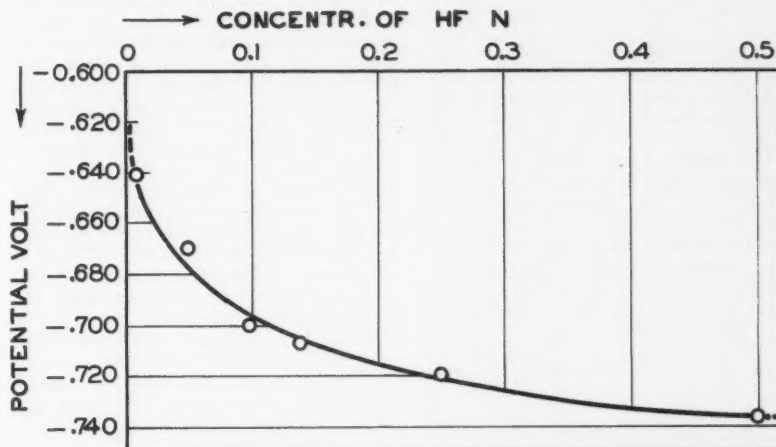


Figure 7—Steady potential of Zr (see Figure 6) and concentration of HF .

TABLE 2— d -values in \AA and Relative Intensities (I/I_0) of Diffraction Lines Obtained From a Passivating Salt Film on Zr in a KF Solution*

SAMPLE		STANDARD PRINCIPAL d VALUES		
d in \AA	I/I_0	K_2ZrF_7	$KF \cdot 2H_2O$	I/I_0
4.43	53	...	4.42	63
3.15	100	3.18	...	100
2.96	100	...	3.01	100
2.60**	2.58	75
2.24	35	2.24	...	50
1.83	35	1.83	...	50

* Zr lines omitted. Comparison with the ASTM cards.

** Zr line.

As the film grew in thickness the pores became smaller in size with the potential slowly becoming even more positive. Simultaneously the rate of dissolution, which occurred in these narrowing pores, passing a more or less flat maximum, gradually decreased.

At the 75th minute the fluoride was added. As already described and shown in Figure 5, the black (hydride) film breaks down, partially exposing the

bright unprotected metal to the action of the acid. As a consequence the potential dropped at once to values as low as -1.076 volt and the rate of dissolution of the unprotected areas of the metal increased simultaneously (direct dissolution, not through the pores). However, because of the increase of $[Zr^{4+}]$ in the interface, reaction (4) occurred with the result that insoluble salts started to crystallize on the Zr , blocking its surface

TABLE 3—Solubilities of Fluozirconates at 20 C.

Fluozirconate	Solvent	Solubility (moles/l)
$(\text{NH}_4)_2\text{ZrF}_7$	H_2O	0.55 ²⁰
$(\text{NH}_4)_2\text{ZrF}_6$	H_2O	1.05
K_2ZrF_6	0.10N HF	0.074*
K_2ZrF_6	H_2O	0.053 ⁴
K_2ZrF_6	0.125N HF	0.0655 ²⁰
K_2ZrF_6	5.890N HF	0.1287
Na_2ZrF_6	0.10N HF	0.016*

* The saturated solutions (25C) were complexed with versine and the excess of the latter was titrated back with a bismuth nitrate solution.

again. Hence, the potential rose more or less slowly and the rate of dissolution decreased even to complete passivation in the case of NaF additions.

From the section of this article entitled "Passivating Films on Zr," it follows that the salt films formed consist of fluozirconates. The effectiveness of NH_4F , KF and NaF additions in passivation may then be explained by the relative solubilities of fluozirconates. Table 3 gives data for solubilities of fluozirconates that were available in the literature, or were obtained in the laboratory. Although the table is incomplete it nevertheless provides a basis for some qualitative interpretation.

Table 3 shows that the normal fluozirconates (R_2ZrF_6) decrease in solubility in the order NH_4^+ , K^+ and Na^+ . The hepta fluozirconates, R_3ZrF_7 , have the same order of decreasing solubilities, although they may not be quite stable in air.²³ The very limited solubility of sodium fluozirconates is the reason why the potential of Zr (Figure 8) at the 75th minute does not drop as low as in the case of NH_4F and KF additions: the passivating salt layer, because of the low solubility of the sodium fluozirconate (see Table 3) begins to form so fast that after the breakdown of the hydride film, the moment of lowest potential could not be caught by the experimental arrangement used. It is also possible that both films partially overlap.

Figures 2, 3 and 4 show that the rates of dissolution of Zr at first increase upon additions of fluorides to the acid. This increase may be explained by the increase of the conductivity of the acid¹⁹ as well as by the shift in equilibrium to undissociated HF. The decrease at higher

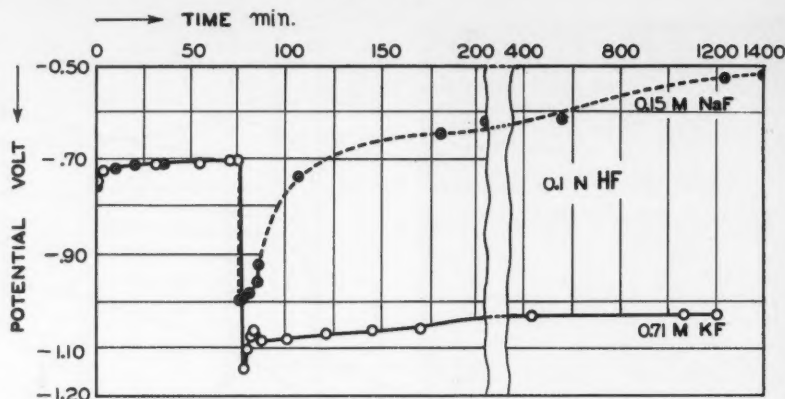


Figure 8—Potential-time curves of Zr in 0.1N HF showing the effect of addition of NH_4F or of KF (solid curve) and of a 0.15M NaF solution (dashed curve) after 75 minutes of dissolution in pure 0.1N HF.

concentration of fluorides is then attributed to the formation of salt films.

Concerning the NH_4F additions there is a plot of solubility of $(\text{NH}_4)_2\text{ZrF}_7$ versus NH_4F concentrations published by von Hevesy.²⁰ This plot shows that the solubility of $(\text{NH}_4)_2\text{ZrF}_7$ decreases sharply in NH_4F solutions up to 4M, and remains constant after that point even up to 10M NH_4F . In reference to Figure 2 it can be seen that the rate of dissolution of Zr began to decrease in NH_4F solutions over 3M, which is the point when the $(\text{NH}_4)_2\text{ZrF}_7$ becomes least soluble in NH_4F solution. However, this solubility is still high enough not to produce a completely continuous salt film on the Zr surface. Thus, NH_4F additions to HF will not cause a perfect passivation of Zr. The rates of dissolution in 6M and 8M NH_4F solutions were nearly the same, indicating that the solubility of the salt film is a critical factor in passivation of Zr in HF by fluoride additions.

No experiments have yet been made to decide whether or not the dissolution of Zr in HF is an electrochemical process.

Acknowledgments

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Any discussion of this article not published above will appear in the December, 1959 issue

Performance of Organic Coatings in Tropical Environments*

By A. L. ALEXANDER, B. W. FORGESON and C. R. SOUTHWELL

Introduction

EARLY IN 1940 plans were in preparation to expand the facilities of the Panama Canal either by constructing a sea-level route or by adding a third set of locks. To insure specification of the optimum materials available, an elaborate program was begun by the Panama Canal Company to develop detailed data describing the corrosion characteristics of a variety of metals and the protective qualities of a considerable number of organic coatings when exposed to the natural environments of the tropics. Initial studies of limited scope were completed and reported.^{1,2,3}

Plans for the canal construction were suspended indefinitely with the onset of World War II. From the data obtained in the initial studies, it was concluded that insufficient samples had been provided, and the exposure periods had been too brief for results of desired reliability and significance. Accordingly, a second program was undertaken from which some results describing the performance of selected metals in the program have been reported.^{4,5}

This discussion is not concerned with the formulation, description and subsequent evaluation of new or experimental products. This discussion is concerned with the establishment of reliable quantitative data, from products available at the beginning of this study, to describe those organic coatings that would be the most satisfactory for protecting metals exposed to the corrosive environments characteristic of tropical latitudes. A few experimental formulations were included, and those selected were based on preliminary data that recommended them for this service.

Addition of the organic coatings study to the existing metals program required a judicious selection of available coating systems with properties considered most adaptable to tropical conditions. For evaluation they were applied to carefully prepared metal surfaces and exposed at the sites described for the metals.⁴ These environments consisted of continuous and mean tide immersion in sea water, continuous immersion in fresh water and continuous exposure to marine and inland atmospheres.

Test Methods Used

Exposure Environments

Any organic coating system displaying marked physical stability and protective qualities when applied to steel and exposed to one or more local environ-

ments was assumed to be of some practical value for service in the Canal Zone. Therefore, in the design of the experiment, sufficient panels were prepared with each system for exposure of duplicates at each location.

The roof of the Washington Hotel in Cristobal provided a marine environment located approximately 300 feet from the water's edge at an elevation of about 55 feet. At the Fort Amador pier the panels were exposed on racks 10 feet above high tide.

For inland exposure, panels were located at Miraflores (5 miles inland from the Pacific coast in a cleared site) and at Gatun at a point about equidistant from the Atlantic Ocean. Panels were mounted on stainless steel racks and held in position by porcelain knobs. The racks were inclined at an angle of 45 degrees and faced south except at Cristobal. At Cristobal the prevailing winds were from the sea; at Fort Amador, winds were more variable but predominantly from the land side. Typical climatic conditions are shown in Figure 1 for both the Atlantic and Pacific sides of the isthmus.

For continuous sea water immersion, test specimens were suspended 14 feet below mean tide elevation from the pier on the Fort Amador causeway at a point 2 miles offshore in the Pacific Ocean. Panels for evaluation at mean tide were placed at this same location in such a position that the tides, which averaged 13 feet daily, caused the panels to be exposed alternately to air and sea water for approximately equal periods. Continuous immersion in fresh water was provided by suspending the specimens from the test pier in Gatun Lake, whose average temperatures are shown in Figure 1.

Abstract

Seven-, six-, three-, and one-year exposures have provided quantitative data on the ability of organic coating systems to protect metals from the five corrosion environments characteristic of tropical latitudes. Data are reported on the performance of more than 350 coating systems in which alkyls, epoxys, synthetic rubbers, phenolics, vinyls and bitumens were used singly or in combinations and mixtures. 2.25

Materials

The systems selected consisted of materials whose merit had been established in less corrosive environments of temperate latitudes. In a few instances, some experimental products were included because preliminary data suggested they possessed unusual merit. Effort was made to include formulations representing all of the more prominent types of resinous matrices commonly associated with corrosion control. Similarly, those pigments ordinarily specified in corrosion control were included, sometimes in several resin combinations. Generally accepted methods of panel preparation and application of materials were followed.

Results of Tests

Visual inspection and rating of atmospheric and immersion panels were made at 1-, 2-, 4- and 6-month intervals and at 1-, 3- and 6-year intervals. An inspection was made of specimens exposed to the atmosphere for 7 years. When a test panel had deteriorated so that the coating was no longer providing satisfactory protection to the underlying steel (a numerical rating of less than 7), the test was concluded and the panel removed from exposure. Definitions of the numerical rating values used to evaluate each panel are given in Table 1.

Results from this study are now complete for 3-, 6- and in a few instances,

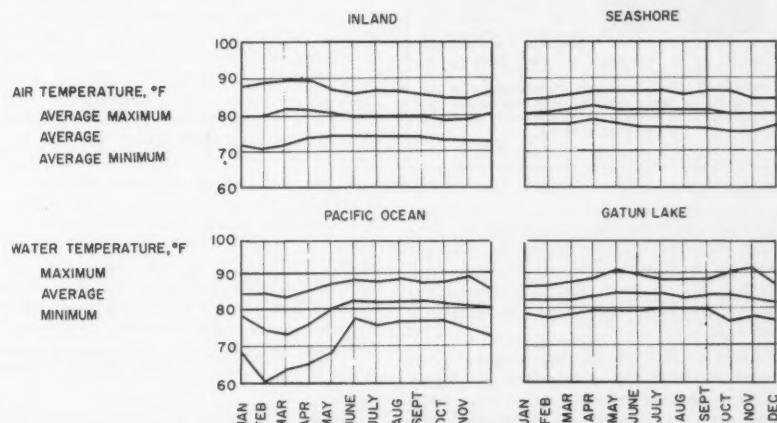


Figure 1—Temperatures for water and air exposures in Panama Canal Zone.

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ALLEN L. ALEXANDER is active in several technical societies, especially in the American Chemical Society and in National Association of Corrosion Engineers. He has held offices in the Chemical Society of Washington (Washington Section, ACS), and in the Paint, Plastics and Printing Ink Chemistry Division of ACS. In NACE he was successively elected secretary - treasurer, 1952; vice-chairman, 1953; and chairman,

1954 of the Baltimore Section. He was chairman of the symposium on Organic Coatings for Tankers at the 1952 NACE National Conference, and a lecturer in NACE short courses at the universities of Tennessee and California.

He also has held offices in the American Society for Testing Materials. Other technical societies of which he is a member are the Federation of Paint and Varnish Production Clubs, the American Institute of Chemists, Washington Academy of Sciences, and Research Society of America (NRL Branch). He is presently an executive in the chemistry branch of the U. S. Naval Research Laboratory and was educated at the University of North Carolina with BS, MS and PhD degrees.

CHARLES R. SOUTHWELL is a Materials Engineer for the Naval Research Laboratory. Since joining the organization in 1954 he has been engaged in the study of deterioration of metals, woods, and protective coatings in tropical environments. From 1950 to 1954 he was Engineer-in-Charge of concrete and protective coatings for the construction of the International Falcon Dam, and prior to 1950 was a Materials Engineer for the Panama Canal Company doing investigative work pertaining to concrete, metals, and protective coatings for use in design of a sea-level canal at Panama. He attended St. Mary's University of Texas, and is a member of NACE.



B. W. FORGESON is in charge of the tropical exposure and laboratory facilities in the Panama Canal Zone for the Naval Research Laboratory. He has been in the employ of the laboratory since 1953, during which time he has been actively engaged in the study of corrosion, protective coatings and the biological deterioration of materials. From 1940 until 1952, he worked for the Special Engineering Division of the Panama Canal as a materials engineer assigned to various research problems and laboratory work concerned with the proposed construction of the third set of locks and the sea level canal. He attended Pennsylvania State University and is a member of NACE.

7-year exposures. This report is concerned only with the long term results. Coating systems that provided satisfactory protection to the sandblasted surfaces for longer periods are shown in Tables 2 through 6.

Only those systems most durable in a given environment are reported. These include those coating systems that gave satisfactory protection (a numerical rating of 7 or better) on all sandblasted

surfaces exposed to a specific environment.

For example, an immersed coating to be included must not have failed on either the scribed or unscribed surfaces. For a system exposed to the atmosphere, the coating must not have failed on any one of eight sandblasted surfaces (scribed and unscribed on the skyward side, and scribed and unscribed on the groundward side at both the Atlantic and Pacific exposure sites).

Three- and six-year results for continuous and alternate sea water exposures are reported with seven-year results for marine atmospheric exposures. Because of the advanced deterioration of most systems in these environments, these results are considered final. Several systems exposed to fresh water and the inland atmosphere are still performing adequately.

Immersion in Sea Water

Eighty-four organic coating systems were evaluated by immersion in sea water at Fort Amador. Top coats of 45 systems were classified as antifouling paint. Vehicle types include 1 alkyd, 2 epoxies, 5 synthetic rubbers, 11 phenolics, 14 vinyls, 23 bitumens, and 28 combinations and/or mixtures of these. From this total, 24 maintained a satisfactory rating on the scribed and unscribed surfaces during 3 years' exposure. These consisted of the following: 12 hot applied bitumens, 7 vinyls, 2 phenolics, 2 synthetic rubbers and, somewhat surprisingly, 1 alkyd. Antifouling top coats were applied to 16 of the 24 systems.

After six years, only three systems (1368B, 1370B and 5670B) appeared to protect the metal adequately, each of which was in remarkably good condition. The three systems listed in Table 2 maintained adequate protection of the metal for the exposure period, and the films themselves were physically intact at the time of observation.

The three outstanding coating systems were applied at relatively heavy film thickness. Among the comparatively successful materials were the vinyls which performed adequately for 3 years, although the films were applied at thicknesses greater than ordinarily associated with vinyls. Adequate applications of coal-tar primer and enamel, followed by a reliable antifouling paint, apparently will provide a highly successful system for protection of metal against tropical sea water corrosion. This protection can be accomplished with fewer coats than with a synthetic rubber system although the latter can be expected to give equivalent results. The coal-tar-antifouling system affords remarkable protection from undercutting at the scribe mark. Differences here are more marked in the case of the vinyl systems although in one case the unscribed panels failed more severely than the scribed one. These data give little evidence to indicate a strong preference for wash primer over a phosphoric acid wash in prolonging the life of either system.

Of the remaining 16 systems performing reasonably well after 3 years, coal-tar

systems and competitive vinyls are prominent. Bitumens and vinyls, representative of two classes of materials, offer excellent prospects for protection in this kind of service. However, 3-year protection can be expected from a properly formulated phenolic or alkyd resin system coupled with a high quality antifouling paint. In any case, length of protection is proportional to film thickness, and the matrix material must be adaptable to application in heavier films.

Mean Tide Exposure

Of the exposure environments studied, failure at mean tide was the most severe. Of the 83 systems in this phase of the investigation, only five were performing adequately after 3 years, and none were considered entirely satisfactory for the full 6-year period. This compares with 24 and 3 systems at 3 and 6 years respectively in continuous immersion. Of the 83 systems studied at mean tide, matrices were as follows: two alkyds, two epoxies, five synthetic rubbers, 11 phenolics, 20 bitumens and 28 combinations and/or mixtures of these.

Data on the five systems considered adequate after 6 years are shown in Table 3. Intermittent exposure to sunlight and air, in addition to sea water eliminated rapidly those coatings other than the vinyls and two synthetic rubbers. This is true in spite of the fact that several bitumen systems were applied at greater thicknesses. For tide level service, the vinyl systems including a top coat of antifouling paint offer greater protection. If thicker films are permissible and no antifouling requirements are implied, either of two synthetic rubber systems are acceptable. Based on the systems reported in Table 3, there is no marked difference in paint behavior on scribed and unscribed panels. The wash primer pretreatment of steel before application of vinyl primer is considered standard procedure.

Fresh Water Immersion

The fact that four systems immersed in fresh tropical water of Gatun Lake remained in almost perfect condition for the 6-year period indicates that many more coating systems are available for satisfactory service in this environment than in either of the others discussed to

TABLE 1—Description of Visual Rating System

Numerical Value	Condition of Coating and Panel
10	Perfect (no deterioration of any kind)
9	Slight deterioration of coating
8	Intermediate (between 7 and 9)
7	Deterioration evident with a few defects in coating; continued protection of underlying metal; coating considered satisfactory.
6	Moderate deterioration, some coating defects, slight corrosion of underlying metal surface. Some surface preparation and repainting necessary for continued protection. Coating considered unsatisfactory.

this point. At the end of the 6-year exposure, 30 out of 64 systems studied were protecting the metal substrates quite well. The 64 vehicles included one alkyd, two epoxys, four synthetic rubbers, 13 vinyls, 15 bitumens, 16 phenolics and 13 combinations and/or mixtures of these. The survivors were four phenolics, three synthetic rubbers, ten vinyls, one alkyd, eight bitumens and five combinations. The four systems remaining essentially perfect during the exposure, as shown in Table 4, consist of one vinyl, one bitumen and two synthetic rubber systems. Several vinyl and coal-tar type coatings are available which can be expected to give good service in fresh water. With bitumens and synthetic rubbers, greater film thickness is required than with the vinyls. No marked differences were apparent to distinguish between the value of wash primer and phosphoric acid pretreatment.

Marine Atmosphere

Only 10 out of 64 systems tested in the marine atmosphere were considered adequate after 6 years as compared to 30 out of 84 systems exposed to fresh water. This indicates the severity of the tropical marine atmosphere. An additional 1-year exposure eliminated six of these. Only four systems were performing acceptably after 7 years. This additional 1-year exposure was believed to be sufficient to indicate a marked difference in protective qualities of two finish classes adaptable to this particular environment. Vehicles used in the 64 systems consisted of three alkyds, four synthetic rubbers, nine phenolics, 13 vinyls, 15 bitumens and 20 combinations or mixtures of these.

As given in Table 5, an alkyd primer pigmented with red lead was included in the first four finishing systems which provided adequate protection to both scribed and unscribed surfaces and to the skyward and groundward sides of each panel. With a single exception, each primer was followed by an alkyd or phenolic top coat, the exception being an asphalt emulsion. Thinner films were required than for emulsion or bitumen-type materials. In this latter case, coal-tar or bitumen-type coatings afforded good protection for 6 years but began to show degradation after an additional year. This would indicate the choice of alkyd-red lead primers followed by top coats of the more durable enamels based on either alkyd or phenolic vehicles for marine atmospheric service in the tropics.

Data of Table 5 indicate that systems including red lead primers provide better protection against undercutting along scribe marks than do the coal-tar primers. This same difference is not so apparent in the immersion tests. No marked differences appeared between groundward sides as compared to skyward sides of the alkyd-phenolic panels. Some differences did appear on the coal-tar systems although there is little consistency in either direction. Thus, a good primer, properly pigmented with an anticorrosive pigment, is preferable to the heavier coal-tar type coating for protection against undercutting corrosion along a film break. On the other hand, each type finish stands up equally well to either groundward or sky-

TABLE 2—Sea Water Immersion

System	Thickness (mils)	No. of Coats in System	SERVICE RATING			
			3 Years		6 Years	
			Unscribed	Scribed	Unscribed	Scribed
1368B*	80-100	1-Coal-Tar Primer 2-Coal-Tar Enamel 3-4 Natural Resin A-F†	10	10	9	8
1370B*	70-85	1-Coal-Tar Primer 2-Coal-Tar Enamel 3-4 Natural Resin A-F**	10	10	8	8
5670B*	40-55	1-Synthetic Rubber (Neoprene) 2-13 Synthetic Rubber (Neoprene)	10	10	8	9
1260B*	41	1-2 Alkyd Primer 3-Hot Plastic A-F	10	10	0	1
1367B*	85-115	1-Coal-Tar Primer 2-Coal-Tar Enamel 3-Hot Plastic A-F	10	10	7	5
1457D†	8.5	1-3 Vinyl Primer†† 4-5 Vinyl A-F††	10	10	7	5
1458D†	18	1-2 Vinyl Primer†† 3-8 Vinyl A-F††	10	10	3	6
4567B*	18-27	1-2 Vinyl Primer 3-10 Vinyl Enamel	10	9	9	5

* Phosphoric acid pretreatment.

† Wash primer pretreatment.

‡ Navy cold plastic A-F paint.

** Experimental NRL formula.

†† Similar competitive systems.

TABLE 3—Mean Tide Exposure

System	Thickness (mils)	No. of Coats in System	SERVICE RATING			
			3 Years		6 Years	
			Unscribed	Scribed	Unscribed	Scribed
1236D*	7.5	1-Vinyl Primer 2-Vinyl A-F	7	8	2	2
1457D*	8	1-3 Vinyl Primer 4-5 Vinyl A-F	10	10	5	5
1458D*	18	1-3 Vinyl Primer 4-8 Vinyl A-F	10	10	6	6
5680B†	2-250	1-3 Synthetic Rubber	8	9	5	5
7890A	25-35	1-Synthetic Rubber	10	10	7	5

* Wash primer pretreatment.

† Phosphoric acid pretreatment.

TABLE 4—Fresh Water Immersion*

System	Thickness (mils)	No. of Coats in System	Service Ratings After 6 Years	
			Scribed	Unscribed
4679D†	2.5	1 Vinyl Enamel	10	10
2456B‡	38-60	1 Coal-Tar Primer 2 Coal-Tar Enamel	10	10
5670B‡	38-52	1-13 Synthetic Rubber	10	10
7890A	32	1 Synthetic Rubber	10	10

* Of 64 systems tested in fresh water, 30 systems including a variety of vehicle types, were satisfactory. The four systems shown here were in excellent condition after 6 years.

† Wash primer pretreatment.

‡ Phosphoric acid pretreatment.

ward exposure. Synthetic rubbers and vinyls are conspicuously absent among the survivors listed in the marine atmosphere. However, these types might be expected to yield more rapidly to the intense solar radiation of the tropics.

Inland Atmosphere Exposure

Materials identical to those exposed to marine atmosphere were exposed also to the relatively clean inland atmosphere of the Canal Zone. A summary of these

results is given in Table 6. The survival rate was greater than the marine environment. Ten of the systems were performing adequately after 7 years' exposure. These systems are identical to those which survived a 6-year marine exposure.

A notable addition is the single synthetic rubber coating (5670B). Of additional interest is the relatively stronger position of the bitumens here when compared with the alkyds or phenolic systems. The presence of the anticorrosive pig-

ment is not so essential for protection against inland exposure. Further, several coal-tar systems were included which performed well when applied at reduced film thicknesses as compared to that required for immersed service. There appears to be little difference in ability of either type coating to prevent undercutting of the film along the scribe mark under inland exposure.

Summary

No general purpose coating system is available among those studied. On the other hand, the data indicate that for each of the environments described there were one or two general classes of materials particularly adaptable to each specific condition.

In sea water, for example, coal-tar systems offered acceptable protection up to 6 years. In the same environment the vinyls provided excellent service through 3 years; most of the other type finishes studied failed. In view of the relative thin films which characterize the vinyls, a convenient choice of two systems is offered, and the final selection may be made on the basis of length of service required. Little or no difference is apparent in the ability of each system to prevent undercutting of the film along the scribe mark.

Of the five environments studied, mean tide exposure was most severe, and for such service the vinyls and synthetic rubbers offered the best protection. Probably the alternate drying and wetting, plus intermittent exposure to air, degraded the other coating types more rapidly.

For fresh water service, a greater coating selection appeared acceptable. Coal-tar enamel and vinyl or synthetic rubber protected steel perfectly in fresh water during a 6-year period, and a large number of other materials were considered adequate throughout the experiment.

Synthetic enamels based on alkyd and phenolic resins were preferable for marine atmospheres. The coal-tar systems, however, were quite efficient but required greater thicknesses. The synthetic enamels, however, appeared to check undercutting along the scribe marks better than the coal-tar enamels.

Similarly, enamels based on coal-tar and alkyd-phenolic resins provided outstanding protection in an inland atmosphere. When removed from the salt-laden atmosphere, undercutting along a break in the film was controlled about equally with each system.

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TABLE 5—Marine Atmosphere

System	Thick-ness (mils)	No. of Coats in System	SERVICE RATING							
			6 Years				7 Years			
			Skyward		Groundward		Skyward		Groundward	
			U†	S**	U†	S**	U†	S**	U†	S**
2347B*	7-20	1-2 Alkyd-Red Lead..... 3-4 Asphalt Emulsion	7	7	8	8	7	7	8	8
3478B*	10	1-Alkyd-Red Lead..... 2-4 Alkyd Enamel	8	8	8	7	8	7	7	7
3590B*	3	1-2 Alkyd-Red Lead..... 3-4 Phenolic Enamel	8	8	9	9	8	7	9	8
3590B†	3	1-2 Alkyd Primer..... 3-4 Phenolic Enamel	8	9	9	8	8	7	9	7
2349B*	54	1-Coal-Tar Primer..... 2-3 Coal-Tar Enamel	8	7	9	8	6	3	7	4
2350B*	54	1-Coal-Tar Primer..... 2-3 Coal-Tar Enamel 4-Cumar Aluminum	9	8	9	9	8	4	9	3
2370B*	7-12	1-Coal-Tar Enamel..... 2-Coal-Tar Emulsion	9	8	8	9	7	5	6	4
2379B*	5-30	1-Coal-Tar Primer..... 2-3 Coal-Tar Emulsion	10	7	9	7	8	3	7	3
2380B*	56	1-Coal-Tar Primer..... 2-Coal-Tar Enamel	9	8	10	9	6	4	7	5
3450B*	2-15	1-Phenolic-Primer..... 2-Resin and Asbestos Filter	8	7	7	7	6	4	6	4

* Phosphoric acid pretreatment.
† Wash primer pretreatment.

† Unscribed.
** Scribed.

TABLE 6—Inland Atmospheric Exposure

System	Thick-ness (mils)	No. of Coats in System	SERVICE RATING							
			6 Years				7 Years			
			U**	S††	U**	S††	U**	S††	U**	S††
2348D*	4-15	1 Phenol-Alkyd..... 2-3 Asphalt Emulsion	8	8	9	8	8	8	9	7
2368B†	5-20	1-2 Coal-Tar Emulsion.....	8	8	8	8	7	7	7	8
2370B†	3-13	1-2 Coal-Tar Emulsion.....	9	10	9	10	8	9	7	10
2378B†	7-15	1-2 Coal-Tar Paint.....	9	9	9	9	8	9	9	8
2379B†	10-25	1 Coal-Tar Primer..... 2-3 Coal-Tar Emulsion	10	10	10	10	10	10	10	9
2450D‡	10-40	1-2 Coal-Tar Primer..... 3 Asphalt Paint	8	8	9	9	7	7	9	9
3478B†	10	1 Alkyd Primer..... 2-4 Alkyd Enamel	9	9	10	9	9	7	10	7
3590B†	3	1-2 Alkyd Primer..... 3-4 Phenolic Enamel	9	8	9	8	9	7	9	7
3590D*	3	1-2 Alkyd Primer..... 3-4 Phenolic Enamel	8	7	9	8	8	7	9	7
5670B†	34-42	1-13 Synthetic Rubber..... (Neoprene)	9	9	8	8	9	9	9	9

* Wash primer pretreatment.
† Phosphoric acid pretreatment.
‡ Proprietary phosphate wash.

** Unscribed.
†† Scribed.

Any discussion of this article not published above
will appear in the December, 1959 issue

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Stress Corrosion Crack Paths in Alpha Aluminum Bronze In Ammonia and Steam Atmospheres*

By J. F. KLEMENT, R. E. MAERSCH and P. A. TULLY

Introduction

MANY OF the metallurgical factors involving causes and mechanisms of stress corrosion cracking in some alloy systems can be associated with the path followed by the propagating corrosion cracks. A series of stress corrosion tests on aluminum bronze alloys has resulted in a general basis for the diagnosis of field failures under corrosive conditions. This could be a valuable tool for research into corrective measures of alloy modification and processing procedures which will tend to reduce service stress corrosion cracking failures to a minimum.

The tests involve the use of two characteristically different media which have been associated with field failures in certain applications of alpha aluminum bronze alloys. The corrosive agents involved are:

1. Steam, which can cause stress corrosion cracking in aluminum and/or silicon bronze alloys under conditions of high tensile stress and temperature.
2. Ammonia vapor, the presence of which has long been associated with stress corrosion cracking in many copper alloys, including aluminum bronze.

The observations concerning crack paths are explained by known crystallographic phenomena. Of major importance in the explanation of crack paths is the phenomena of equilibrium grain boundary segregation, wherein a relatively high concentration of solute atoms tends to build up in grain boundaries under both commercial production and equilibrium conditions.

Test Procedure

Materials

Tests were conducted using specimens taken from three different aluminum bronze alloys which are available in 1/4-inch plate from commercial sources. Two of the alloys were binary copper-aluminum alloys containing a nominal concentration of 5 percent and 8 percent aluminum. The third alloy was a ternary alloy containing a nominal 6.5 percent aluminum and 2.5 percent iron. A careful chemical analysis of the alloys, which by various specifications are allowed 0.5 percent total impurities, showed the alloys to be of satisfactory purity for the tests. The analysis of the alloys is given in Table I.

The problem of selecting the condition of the materials for test was not easily resolved. The difference in analysis



JOHN F. KLEMENT has been Technical Director, Ampco Metal, Inc., since 1951. He formerly was chief metallurgist and wrought products superintendent. He has a BSME, Marquette University, 1940.

R. E. MAERSCH—Chief Development Metallurgist, Ampco Metal, Inc., Milwaukee. He received a BS in chemical engineering, University of Wisconsin, 1937, after which he worked for Gisholt Machine Co. He also worked on quality control in the aircraft division of J. I. Case Co., Rockford, Ill. He belongs to ASM, AWS and NSPE.



PATRICK A. TULLY—Wrought Products research metallurgist for Ampco Metal, Inc., Milwaukee, where he has been employed since graduation in 1951 from University of Wisconsin. He has an MS in metallurgy from Wisconsin, 1955. He is a member of ASM and an instructor in metallurgy in the evening division of the University of Wisconsin in Milwaukee.

of the alloys produced the expected differences in inherent properties. Added to this, differences in recrystallization temperatures, grain growth characteristics, and work hardening rates made the selection of a "condition parameter" difficult.

Preliminary testing indicated that the variables did not have a major effect on crack paths, and therefore the practical parameter of tensile strength was selected. The tensile strength of the 95-5 alloy and 92-8 alloy was approached by cold rolling from an annealed structure since the alloys were supplied in the "1/2 Hard Temper." The 90-6.5-2.5 alloy was annealed down to the strength level

Abstract

The investigation presented illustrates the importance of analyzing stress corrosion crack paths, and the interpretation of the crack paths with respect to alloys and corrosive media. Testing involved alpha aluminum bronze alloys of 5-8 percent Al content exposed to ammonia and steam atmospheres, while stressed to about 80 percent of the yield strength. The various crack paths obtained are illustrated in micrographs, and the reasons for the different types of crack paths are associated with factors such as equilibrium grain boundary segregation and the chemical attack of segregated solute elements by corrosive agents. The results of the investigation are discussed in terms of analyzing causes of field failures. Examples of processing considerations to minimize failures are cited. 3.2.2

after hot rolling. The strength parameter was selected from a use viewpoint, in order to compare alloys in the condition in which they may be used commercially in a similar application.

Specimen Preparation

Test strips 1/4-inch x 5/8-inch wide x 2 5/8 inches long were sawed from the plates, and milled on one side to 1/8-inch thickness. These strips were then mounted in stainless steel fixtures* designed to bend the strips in the manner of end supported—center loaded beams by means of a center screw, with the 5/8-inch wide "as received" surface of the specimen exposed under tension to the corrosive. The specimens were then bent to a center deflection to produce a fiber stress of about 30,000 psi.

Steam Atmosphere Tests

The tests in steam were carried out in pressure-tight stainless steel cylinders which held the stressed specimens and a supply of water. The specimens were suspended over the water by means of a perforated plate. The cylinders were charged into a 350 F electric oven, where they remained for test periods of two days after reaching testing temperature.

At the end of these periods, specimens were examined for surface cracks. Specimens which did not crack were recharged into the cylinders for additional two day exposure periods. This method of exposure and inspection was continued until all specimens showed signs of cracking. They were then removed from the fixtures and prepared for metallographic examination.

* Preliminary testing using aluminum bronze stressing fixtures and stainless steel fixtures with and without insulating pads to isolate the specimens revealed that the results were unaffected by galvanic phenomena in the atmospheres and temperatures used. The use of stainless steel fixtures without insulation was adopted because the aluminum bronze fixtures themselves sometimes became victims of stress corrosion cracking after several test runs.

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Figure 1—Intergranular cracking in 5 percent Al, 95 percent Cu alloy stressed at 30,000 psi in 350 F steam atmosphere. 100X.



Figure 2—Intergranular and transgranular crack path in 5 percent Al, 95 percent Cu alloy stressed at 30,000 psi in room temperature ammonia atmosphere. 150X.



Figure 3—Intergranular cracking in 6.5 percent Al, 2.5 percent Fe, 91 percent Cu alloy stressed at 30,000 psi in 350 F steam atmosphere. 50X.

Ammonia Atmosphere Tests

Ammonia atmosphere tests were carried out under a bell jar at room temperature. The specimens, stressed in fixtures, were suspended over a beaker containing 10 percent ammonium hydroxide solution. The bell jar was sealed against leakage except for a capillary tube protruding from the side. Specimens were exposed for a period of three days and then removed and prepared for metallographic examination.

Limited additional tests were conducted using an ammonia atmosphere of 350 F in order to study the ammonia crack paths formed at this temperature. The object of this test was to confirm that the crack paths observed in the primary work was dependent on the type, but not the temperature, of the corrosive medium.

It should be noted that no effort was made to evaluate the ammonia cracking susceptibility with respect to failure time during these tests. To facilitate metallographic crack studies it was necessary to terminate tests while most of the specimens were in the stage of incipient cracking. Since the exact time of crack beginning is virtually impossible to determine, and no failure times were determined, relative crack severity at the time of test termination had to be used as a substitute for these quantitative data.

Metallographic Examination

The center portion was cut from the strips after their removal from the fixtures. These were mounted in bakelite, with the $\frac{1}{8}$ -inch edge transverse to the cracks exposed for polishing. Approximately $\frac{1}{16}$ -inch was ground from the edge, and the specimens were polished and etched with 5 percent potassium dichromate for examination.

Crack paths were determined by observations at a number of different magnifications. Micrographs of examples of

TABLE 1—Analysis of Test Alloys

Alloy	ELEMENT, PERCENT					Tensile Strength, Psi (ave.)
	Cu	Al	Fe	Ni	Mn	
92-5.....	94.53	5.44	.03	65,000
92-8.....	91.64	8.25	.02	65,000
90-6.5-2.5.....	91.12	6.48	2.28	.10	.02	75,000

TABLE 2—Characteristic Crack Paths in Specimens (30,000 psi stress level)

Alloy (Bal. Cu.)	Corrosive	Temperature	Crack Description
5% Al.....	Steam	350 F	Intergranular
	Ammonia	Room Temperature	Random
6.5% Al; 2.5% Fe.....	Steam	350 F	Intergranular
	Ammonia	Room Temperature	Transgranular
8% Al.....	Steam	350 F	Intergranular
	Ammonia	Room Temperature	Transgranular
6.5% Al; 2.5% Fe.....	Ammonia	350 F	Transgranular
	Ammonia	350 F	Transgranular

each crack were taken at magnifications and locations which typically illustrated the cracks. A description of crack paths is given in Table 2.

Results

The micrographs in Figures 1 through 6 illustrate the crack paths in the various specimens. In cases where the cracking was rather severe, the path of the principal crack was somewhat difficult to define, and observations were based on the branch cracks. In all cases the fiber stress in the specimens was about 30,000 psi.

Discussion

Steam Cracking

All of the evidence gathered on stress corrosion cracks in alpha aluminum bronze alloys indicated that the cracks

resulting from exposure to steam are intergranular, as are the cracks formed by exposure to hot aqueous solutions and vapors of sea water, acetic acid, sulfuric acid, and sodium hydroxide. General observations on relative failure times during the tests also have indicated that the intergranular cracking susceptibility increases with increasing aluminum content. As an example, the steam test resulted in typical failure times of about 300 hours for the 5 percent aluminum alloy, 150 hours for the 6.5 percent aluminum alloy, and 50 hours for the 8 percent aluminum alloy.

These observations can be used to support a proposed mechanism of the formation of the stress corrosion cracks. This mechanism involves the equilibrium segregation of solute atoms at the grain

Figure 4—Al, 2.5 percent Fe, 91 percent Cu alloy stressed at 30,000 psi in 350 F steam atmosphere. 50X.

boundary. The mechanism proposed by McLean is that the grain boundary is exposed to the atmosphere, and the atmosphere num rate of corrosion is increased by the segregation of solute atoms to the boundary. The segregation of solute atoms to the boundary is a result of the continuous diffusion of solute atoms from the grain interior to the boundary. The diffusion of solute atoms to the boundary is a result of the continuous diffusion of solute atoms from the grain interior to the boundary.

ing water, which is likely to be a result of the failure of the boundary. The failure of the boundary is a result of the continuous diffusion of solute atoms from the grain interior to the boundary.

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Perry's test of the dependence of the rate of oxidation on the concentration of the drosside.

This work on intergranular cracking with the case of oxidizing atmosphere. Many pure corrosion products are two different types of the lattice potentials, and the cracking.



Figure 4—Transgranular crack path in 6.5 percent Al, 2.5 percent Fe, 91 percent Cu alloy stressed at 30,000 psi in room temperature ammonia atmosphere. 250X.



Figure 5—Intergranular crack path in 8 percent Al, 92 percent Cu alloy stressed at 30,000 psi in 350 F steam atmosphere. 50X.



Figure 6—Transgranular cracking in 8 percent Al, 92 percent Cu alloy stressed at 30,000 psi in room temperature ammonia atmosphere. 90X.

boundary of the alloy, as proposed by McLean and Northcott.¹ When the alloy is exposed to the attack of steam or other atmospheres capable of oxidizing aluminum rather rapidly (but incapable of corroding the aluminum bronze rapidly), the segregated aluminum atoms are chemically attacked and form a protective film, normally quite impervious to further attack. However, in the presence of tensile stress, the film ruptures continuously as it forms, and the crack is allowed to propagate along the aluminum rich grain boundary. The identity of the corrosion products (or film) is not known, but the formation of both oxides and hydroxides of aluminum is possible. Preliminary tests in 212 F boiling water and 500 F air yielded results which support the hydroxide as the most likely corrosion product. The tests in air failed to produce any failure or grain boundary penetration over an extended time. The tests in boiling water produced intergranular failures after several weeks. An analysis of the water after the tests indicated the presence of aluminum hydroxide, but no sign of insoluble aluminum oxide, and no detectable copper concentration.

Perryman² has indicated that the extent of grain boundary segregation is dependent upon the degree of saturation of the solvent lattice by the solute. This would serve to explain the increased intergranular stress cracking susceptibility with increased aluminum contents in the case of exposure to steam or other oxidizing media.

Ammonia Cracking

Many investigators have found that pure copper is not susceptible to stress corrosion cracking in ammonia, and that two different atoms must be present in the lattice to produce the electrode potentials required to cause stress corrosion cracking.³ Thompson and Tracy⁴ have

investigated this quantitatively, and shown that at 10,000 psi stress level and 2 percent aluminum, the susceptibility to stress corrosion cracking reaches a maximum in copper-aluminum alloys. The fact that the susceptibility decreases as more aluminum is added can be attributed to the equilibrium grain boundary segregation of aluminum. At 2 percent aluminum based on the authors' observations, the ammonia cracks will be intergranular. As the aluminum content is increased, grain boundary segregation of aluminum increases, and because ammonia will attack copper rather than aluminum, the grain boundaries become more and more resistant to ammonia attack. An alloy composition is finally reached where the grain boundaries will no longer be attacked by ammonia, and intergranular cracking will not occur. A close study of some of the cracks in Figure 4 illustrates this condition, where cracks run almost parallel and very close to certain grain boundaries, but do not enter the boundary except to cross it.

Unlike steam cracks, ammonia cracks may be transgranular, illustrating that the crystals themselves have an ammonia cracking susceptibility. In order to determine just how the transgranular cracking susceptibility varies with aluminum content, it would be necessary to conduct tests with single crystals. It is obvious that, at low aluminum contents the intergranular cracking susceptibility exceeds the transgranular cracking susceptibility, and that this condition gradually changes as the aluminum is increased, until the opposite becomes true at high aluminum contents.

In Figure 7, the dotted curve of ammonia cracking susceptibility is taken from Thompson and Tracy's work at a 10,000 psi stress level. The solid curve at 30,000 psi is an estimated susceptibility curve based on the authors' observations and the Thompson-Tracy curve displaced

to compensate for the higher stress. This curve represents a qualitative picture of ammonia cracking susceptibility for the alpha aluminum bronze polycrystalline aggregate, and may be considered as a resultant curve of intergranular and transgranular cracking susceptibility. The area between (a-a) and (b-b) represents the transition of the crack path from intergranular to transgranular, with a net decrease in cracking susceptibility. Above 5 percent aluminum, the transgranular cracking susceptibility obviously decreases with increasing aluminum content. Whether this is true over the entire range from 0 to 8 percent aluminum is not known, because the shape of the curve is obscured by the more readily occurring intergranular cracking tendency at low aluminum levels, and some statistical composite of the curves in the transition range. It is more likely that the transgranular cracking curve would also exhibit a maximum susceptibility at some aluminum content between 2 percent aluminum and 8 percent aluminum.

The shape and location of the resultant curves would probably be different in each case. The exact locations of vertical lines (a-a) and (b-b) will also be dependent upon the process history of the alloy, and the testing conditions. In order to evaluate susceptibility, curves of this type may be determined on alloys using parameters of equal yield strength, tensile strength and hardness, or after identical processing regardless of properties. The testing stress could be regulated at either a fixed stress level or a percentage of the yield strength of each alloy.

The mechanism of transgranular cracking due to ammonia has been the subject of much speculation and discussion, but no real conclusions have been drawn. Since pure copper does not seem to be susceptible to transgranular cracking, it is obvious that cell action must be an

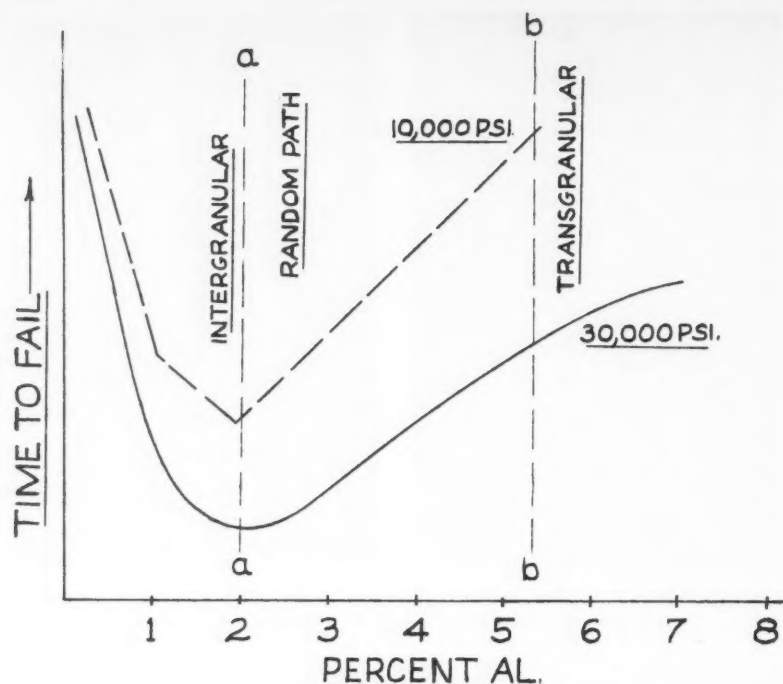


Figure 7—Graphical representation of the stress corrosion cracking susceptibility of alpha aluminum bronze in ammonia atmosphere.

essential part of the mechanism. The corrosion cells within the crystals are easily explained, since it is commonly recognized that alloy crystals are anything but well organized homogeneous lattices. When one considers the total effect of the known phenomena which tend to cause solute atom concentration points in the lattice, it is easy to realize how continuous paths of high corrosion potentials can exist through portions of the crystal. These phenomena include solute atom-dislocation interactions of the electrical, chemical (stacking fault), and physical types,⁹ the recognized clustering of solute atoms around dislocations, polygonization (low angle subgrain) boundaries, disorientations of annealing twin planes, slip planes, and segregation that may be a carry over from the original solidification pattern.

The stress on the crystal is a necessity, but the exact role which it plays is not clear. Stress may be necessary to produce some of the cells through solute atom-dislocation interaction, to open the crack as it forms thus allowing a continued supply of corrosive to propagate the crack, to increase the energy level of the metal at the tip of the crack, or for other reasons not as yet recognized or suspected.

Effect of Iron Content

The stress corrosion cracking susceptibility of the aluminum bronze alloys tested seems for all practical purposes to be independent of the iron content of the alloys. Any small effects that may be found by more careful quantitative work regarding the presence (and amount) of iron would have to be very carefully analyzed as to the proper reasons for these effects. Iron acts as a grain refiner, raises the recrystallization temperature, and adds

to the strength of these alloys. Therefore, if the presence of iron were found to influence stress corrosion cracking susceptibility in some manner, the effect could be of a direct chemical nature (involving the segregation and/or corrosive attack on iron atoms), or of a secondary nature, where the grain size or strength of the alloy resulting from iron additions is the direct cause of susceptibility variations.

Conclusions

In the analysis of field problems concerning stress corrosion cracking in alpha aluminum bronze alloys, the presence of transgranular cracking would indicate that the cracks were caused by the attack of ammonia and/or ammonia bearing corrosives on the alloy.

Careful metallographic examination of cracks in the lower aluminum (approximately 3 percent) alloys should generally reveal some transgranular crack penetration if the cracks were caused by ammonia, even though the crack paths would normally be primarily intergranular in the low alloy range. Where stress corrosion cracks are found to be intergranular, and no transgranular penetration can be found along the main crack or branch cracks, the investigator can generally look to media such as steam, hot acid or caustic vapor, or hot oxidizing solutions for the cause of the cracking. Care must be taken not to confuse intergranular stress corrosion failures with intergranular tensile or creep failures when operating conditions involve temperatures in the range of 500 F or higher.

Since intergranular stress cracking caused by oxidizing agents (steam etc.) appears to be a result of the equilibrium grain boundary segregation of aluminum,

processing steps designed to minimize segregation in alpha aluminum bronze products will aid in minimizing failures if the material must be used in stressed applications. This can be done by avoiding processes which involve high temperature treatments on materials which contain established grain boundaries, a practice which tends to increase equilibrium grain boundary segregation. The use of cold worked and recrystallized material as opposed to hot worked and annealed materials can be recommended in this respect. Cold forming followed by low temperature stress relieving also has been found to be an improvement over cold forming at intermediate temperatures in shaping the alloys.

Avoiding the stress corrosion cracking in the alpha aluminum bronze alloys where exposure to ammonia vapor or solutions is possible is dependent upon the ability of the user to produce a relatively stress free application, since more or less aluminum atom segregation merely effects the intergranular or transgranular cracking tendency. This can be done by stress relief (thermal or mechanical) and proper design. However, the use of these alloys in ammonia applications will always contain an element of risk, since there is never any real assurance that the application of stress relief treatments will eliminate all tensile stresses. Likewise there is no guarantee that operational stresses may not occur through warpage, misalignment of adjacent parts, or expansion and contraction.

Summary

The results of this investigation indicate that equilibrium grain boundary segregation of alloying elements which does not result in the formation of precipitated phases can be considered to be one of the fundamental causes of stress corrosion cracking. The concept can be used to explain the stress corrosion cracking susceptibility of silicon bronze in steam, and the increased resistance of aluminum bronzes to season cracking. Additional study may prove that equilibrium grain boundary segregation and/or the segregation of solute elements around crystallographic faults and subgrain boundaries are the cause of stress corrosion cracking in many of the cases in which there has been no satisfactory explanation as to mechanism.

Additional work on the stress corrosion cracking of alpha phase aluminum bronzes, based on the segregation concept, is now under way. The results to date tend not only to verify the concept, but indicate that it is useful in practical production applications. The progress of this work, along with extensive field testing, will be published as each stage is completed.

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Factors Affecting the Corrosion of Steel By Oil-Brine-Hydrogen Sulfide Mixtures*

By DONALD W. SHANNON⁽¹⁾ and JAMES E. BOGGS⁽²⁾

Introduction

VARIOUS METHODS have been used to reduce hydrogen sulfide attack of oil well equipment. One method, which has been used with considerable success, has been the addition of a chemical inhibitor to the oil at the well-head. In a number of instances, small amounts of these inhibitors (a few parts per million) have reduced corrosion markedly.

The process of finding inhibiting materials has been largely a matter of trial and error. Many of the inhibitors in common use are concoctions of very impure materials about which little is known other than that they do inhibit corrosion. Until the basic nature of chemical inhibition of corrosion are studied more intensively and the mechanisms are more clearly understood, the search for inhibitors must remain one of "hunt and pick."

As the inhibitor industry grew, it became desirable that some standard method of laboratory comparison be set up so that various inhibitors could be compared for their effectiveness. A number of procedures were suggested, including one proposed by a subcommittee of the National Association of Corrosion Engineers.¹ There appear to be serious disadvantages with all of these methods as inhibitor screening tests. The present work was undertaken to evaluate certain of the variables affecting the corrosion process, so that an improved test procedure might be devised.

It seems self-evident that any empirical test method should attempt to duplicate field conditions as closely as is possible under laboratory conditions. Accordingly, all of experiments described here were performed on dynamic systems where the oil-water emulsion flowed constantly over the metal sample.

Experimental Method

In brief, the technique used was a dynamic, weight-loss method, which consisted of placing a weighed piece of steel in a controlled corrosive medium and measuring the loss in weight after a suitable time interval. The containers used were 16 ounce, wide-mouth, "French Square," screw-cap bottles, sealed with a polyethylene gasket. The bottles contained 240 ml of water phase, 240 ml of oil phase, and 30 ml of nitrogen gas. After filling, the bottles were continuously rotated during the course of the experiment at 73 rpm.

Except as noted below, the technique used in the present study was the same as in a proposed new screening test for inhibitors.²

Results

Tests were made to determine whether bottles could be effectively sealed by the technique used, so that H₂S could not escape, and air could not enter during the course of an experiment. The first attempts were made using a fluorocarbon sheet as a gasket inside the bottle caps. Leaks developed when this material proved to be too hard and inflexible. The reaction



proceeds spontaneously in aqueous media, so that whenever a bottle developed a leak, the solution turned cloudy within an hour or so from the free sulfur produced. When a seal of heavy-weight polyethylene film was used in place of the fluorocarbon, no further difficulty with leaks was observed, and free sulfur was never produced.

To make sure that the bottles were air-tight, the following experiment was undertaken. Ten bottles were half filled with the light mineral oil. A solution of H₂S was then made up from oxygen-free distilled water. This solution was forced under pressure through a piece of tubing and introduced underneath the oil layer, so that it never came in contact with air. This solution was added until the oil reached the very brim of the bottle. The plastic film was then held taut over the top (no air was trapped), and the cap was screwed on. Next the bottles were put on the shaker. Bottles were removed periodically, and the water layer analyzed for H₂S by direct titration with standard

Abstract

A study was made of factors affecting the corrosion of steel by oil-brine-H₂S mixtures in order to determine what conditions need to be met in setting up a screening test procedure for possible corrosion inhibitors.

The initial rate of corrosion is dependent on the concentration of H₂S. After the first day, the reaction becomes diffusion-controlled and is independent of H₂S concentration over a wide range. At very high H₂S concentrations, the corrosion rate actually decreases because of a change in the nature of the FeS coating formed on the steel.

The presence of sodium chloride prevents the formation of a protective FeS coating on steel, and the corrosion rate increases with increasing salt concentration, reaching a maximum at about 1 percent salt. Above this concentration, the rate decreases, possibly due to inhibition by the salt itself. 3.4.8

iodine solution using starch for an indicator. The H₂S concentration in the water layer gradually fell as the gas dissolved in oil, reaching a steady-state concentration after 30 minutes. This same value has maintained up to a week, indicating that H₂S was not lost either by leakage out or by reaction with oxygen which leaked in.

Additional evidence for the tightness of the seal is given by the fact that when corrosion test bottles were opened, the sound of escaping hydrogen gas was heard, showing that the seals were holding under pressure.

Polyethylene is affected to some extent by exposure to the oil. It tends to swell slightly, making the seal even tighter. It might be argued that the exposure of any plastic material to the test fluids would give the possibility of introducing inhibitor materials into the system which might interfere with the results. At no time during this work was any inhibiting effect noted from the polyethylene.

To be sure that there was no inhibiting effect from polyethylene, a check

TABLE 1—The Distribution of H₂S Between Water and Hydrocarbon Solvents

Oil Phase	Temp, Degrees C	ppm H ₂ S in Water	ppm H ₂ S in Hydrocarbon	K (Conc. in hydrocarbon / Conc. in water)
Mineral Oil.....	32	77	131	1.70
	32	76	132	1.73
	32	194	334	1.72
	32	196	340	1.73
	32	512	885	1.72
	32	519	889	1.71
	29	1005	1680	1.67
	29	1000	1662	1.66
Benzene.....	31	124	735	5.93
	31	119	708	5.94
	31	125	746	5.96
Kerosene.....	31	221	528	2.39
	31	234	554	2.36
	31	234	577	2.46
Iso-octane.....	31	216	573	2.65
	31	219	579	2.64
	31	215	575	2.67

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⁽¹⁾ General Electric Company, Hanford Atomic Products Operation, Richland, Washington.

⁽²⁾ Department of Chemistry, The University of Texas, Austin, Texas.

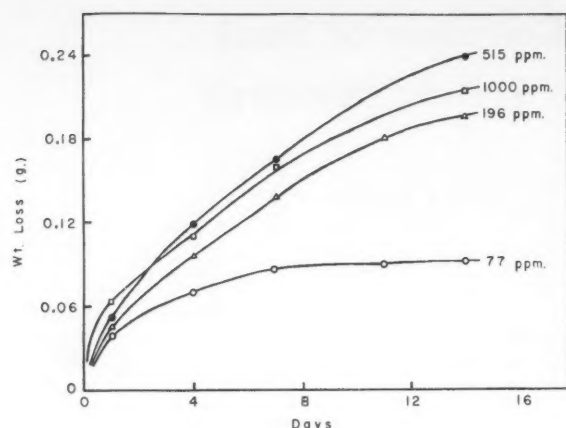


Figure 1—Extent of corrosion as a function of time.

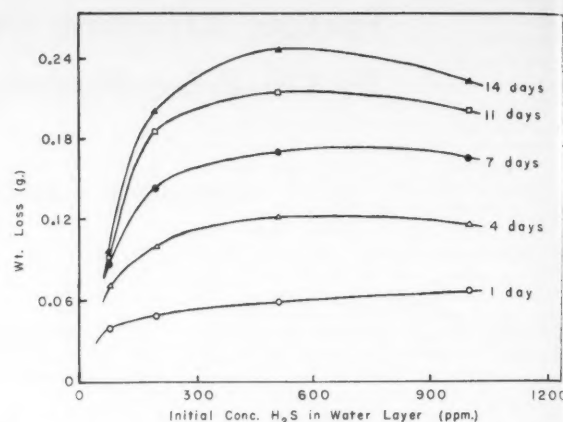


Figure 2—Extent of corrosion as a function of initial hydrogen sulfide concentration.

was run. About 300 square inches of the plastic was cut into pieces and washed with 400 ml of oil for four days on the shaker. This amount of plastic represents about 100 times the surface actually in contact with the oil during a test. Two tests using the standard steel coupons were then run; one using 240 ml of this oil and the other using 240 ml of untreated oil. The same H₂S brine (1350 ppm H₂S, 2 percent NaCl) was used for both. At the end of five days, the first had lost 0.127 gram; the second, 0.125 gram. These values agree within experimental error.

Distribution of H₂S Between Oil and Water

Experiments were next performed to determine the relative concentrations of H₂S in the oil and aqueous phases. Water solutions of H₂S of different concentrations were shaken with mineral oil until equilibrium was established. Both phases were then analyzed for H₂S—the water phase as described above, the oil phase as follows: A 25 ml oil sample was pipetted into a ground-glass stoppered conical flask containing about 15 ml of water and some starch. About 95 percent of the equivalent amount of standard iodine was then added to the flask, and it was stoppered and vigorously shaken. Iodine solution was then added dropwise, with shaking after each addition. When the blue color remained in the water layer, the titration was complete. A blank run with pure oil gave a blue color with one drop of iodine solution.

The results of this series of experiments showed an unexpectedly high concentration of H₂S in the oil phase. In order to ascertain that this was not just a peculiarity of the oil being used, the distribution ratio was determined in the same manner using benzene, kerosene, and iso-octane (2, 2, 4-trimethyl pentane) as the oil phase. All of the results are shown in Table 1. The high solubility of H₂S in hydrocarbon solvents is indeed a general one. This high solubility has not been generally recognized. The implicit assumption that the solubility of H₂S in oil is low shows itself in much of the work described in the literature.

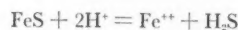
Effect of H₂S Concentration

Next, some experiments were performed to determine the effect of H₂S concentration on the rate of corrosion of steel. The tests were made using the standard technique mentioned above.² Forty-eight bottles were prepared, each containing 240 ml of mineral oil, 240 ml of H₂S solution, and 30 ml of nitrogen gas. In 12 of the bottles, the initial H₂S concentration in the water was 194 ppm; in another set, 622 ppm; in the third set, 1420 ppm; and in the last set, 2560 ppm. The 2560 ppm is a saturated solution at 32 C. Standard steel coupons were placed in 10 of the bottles in each set; the other two contained no metal, and were used to establish the actual H₂S concentration in the water layer after the H₂S became distributed between the two layers. After distribution of the H₂S had taken place, the initial concentrations of H₂S in the water layers were 77, 196, 515 and 1000 ppm, respectively.

Bottles were removed from the shaker after various periods of time, the loss of weight of the steel coupon was determined, and the water layer was analyzed for H₂S. The H₂S analysis was done by direct titration with standard iodine solution, but modifications were necessary in the procedure because the FeS formed as a corrosion product (present as a colloidal precipitate) would interfere. The reaction of H₂S with I₂ is



The HI formed will lower the pH of the solution to the point where the reaction



begins. Both the H₂S released and the Fe⁺⁺ ion will cause errors in the titration. In order to prevent this reaction, it was necessary to buffer the solution strongly at about pH 7. This was easily accomplished by adding several grams of ammonium acetate to the titration flask. After this had been done, the titration of H₂S was done directly using starch as an indicator. In some cases the H₂S content of the oil layer was also determined by the procedure described previously.

The data obtained from these tests are shown in Figure 1, where the weight loss of the coupon is plotted against time, each line representing solutions of different initial H₂S concentration. From this figure it can be seen that the initial corrosion rate is strongly dependent on the H₂S concentration. Qualitative observations also support this conclusion. At the beginning, when shiny metal is placed in the corrosion medium, it takes several minutes for the coupons to turn black in low concentrations of H₂S. At high concentrations they turn black in seconds. At high concentrations, bubbles of hydrogen gas can be seen forming on the surface of the metal.

In the series of experiments at 77 ppm H₂S, there was insufficient reacting material present to maintain corrosion over the period of the experiment. For this reason the weight loss reached a constant value after about a week.

At the three other concentrations, corrosion proceeded continuously throughout the test period. After the first 24 hours, these curves are roughly parallel, showing that the rate of corrosion is independent of H₂S concentration, and apparently depends only on the rate of diffusion of the reacting species through the surface coating of FeS.

The results of the same experiment are shown in Figure 2, where the weight loss of the coupons after varying periods of time is plotted against the initial H₂S concentration in the solution. At low concentrations, the supply of H₂S is exhausted before the experiment is complete, so that the weight loss is less than would be expected. At higher concentrations, the curves are essentially horizontal, showing that the corrosion rate is independent of H₂S concentration in this range.

One very interesting result of the experiments is shown by the 1,000 ppm curve in Figure 1. At first there is a steep rise in the curve, corresponding to the initial rapid corrosion at this high H₂S concentration. Following this, however, the weight loss starts to lag behind, and at the end of two weeks is considerably less than that of the 515 ppm series. Analysis of the water layers showed that

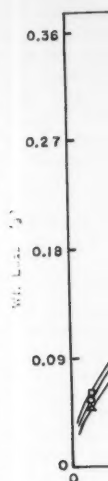


Figure 3—Extent of corrosion as a function of time.

at the end of more than 1,000 ppm series, the corrosion rate was lower than that of the 515 ppm series.

The nature of the conditions under which the high H₂S concentration tests were run is hard to explain, while at the same time, the gelatinous precipitate, the rate to the overall weight loss of the coupons is a supply of more weight in a higher order of magnitude. A rapid corrosion rate is a very

Because of the high concentration of H₂S, the corrosion rate is very high. The test is a very rapid corrosion rate, and the weight loss is very high.

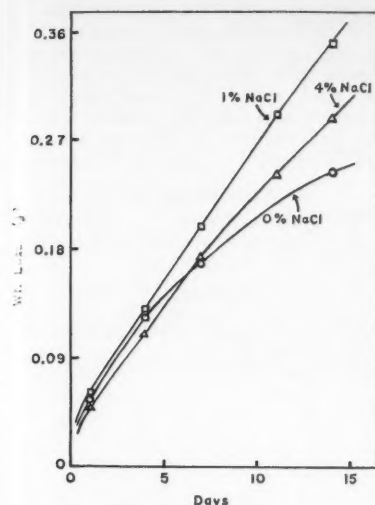


Figure 3—Extent of corrosion as a function of time with various concentrations of added salt.

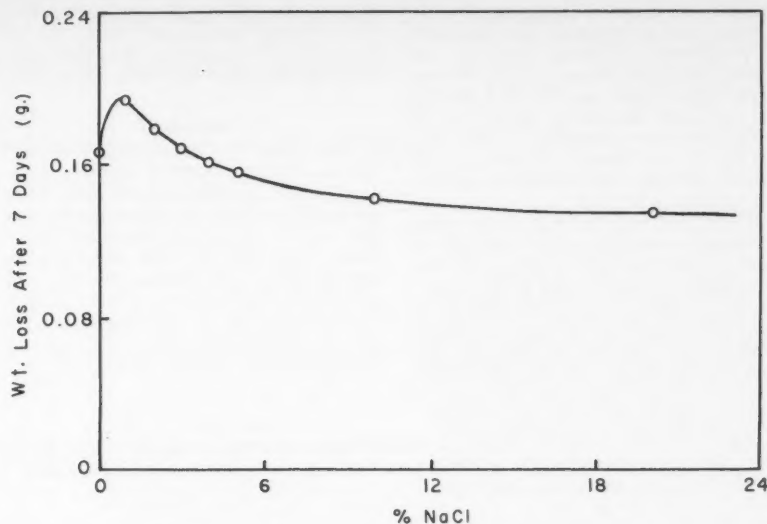


Figure 4—Extent of corrosion as a function of salt concentration.

at the end of two weeks there was still more than twice as much H_2S in the 1,000 ppm series as there was in the 515 ppm series. This indicated that the lowered corrosion rate was not due to any leakage of the bottles or depletion of H_2S .

The nature of the FeS coating formed on the coupons will depend on the conditions under which it was formed. At high H_2S concentration in the solution, the faster initial reaction rate favors small particle size and dense packing of the precipitated particles. At the lower concentrations the slower initial rate gives larger particles which are deposited at a slower rate, and are more subject to being washed off by the agitation of the solution. This difference in the coatings is easily observable on the coupons. At the higher concentration, the coating is hard and crystalline in appearance, while at the lower concentrations it is gelatinous and easily wiped off. As a result, the hard coating slows the diffusion rate to the metal surface, and thus the overall weight loss is less. The implication of this is, that given a continuous supply of corrosive fluid, steel might lose more weight over an extended period of time in a low concentration of H_2S than in a high one. It also points to the danger of drawing conclusions about the overall corrosion rate from the initial rate. A substance which permits initial rapid corrosion, but which favors the formation of a dense, impermeable layer of corrosion product might in the long run be a very effective corrosion inhibitor.

Effect of Salt Concentration

Because salts are present in varying concentrations in all natural waters, it has been the practice to put sodium chloride in the aqueous phase of corrosion tests. Almost universally, when a test is described in the literature, mention is made of the "brine" consisting of a certain percent sodium chloride, with little indication of why the partic-

ular concentration was chosen. Accordingly, the authors have performed a series of experiments to determine the effect of sodium chloride on the corrosion rate.

Two series of ten tests each were run. The bottles were filled as before, except that the H_2S solution contained 1 percent $NaCl$ in the first series and 4 percent $NaCl$ in the second. The H_2S concentration was adjusted to 1500 ± 50 ppm which gives an initial concentration of about 540 ppm in the water layer after distribution between the two phases. The results are plotted in Figure 3.

The first thing that was observed was that the presence of the salt altered the distribution of the FeS precipitate. In the absence of salt, the FeS was present as a fine colloid in the water layer, whereas in even 1 percent salt water, most of the FeS collected at the interface of the oil and water and stabilized an emulsion of water in oil. The excess water, which was not tied up in the stable emulsion, was almost free of FeS .

Also, when any salt was present the hard black coating of FeS failed to form on the steel coupon as it did in the absence of salt. In a static test, the coating formed on the steel, but as soon as agitation was begun, the coating washed off. This indicates that the presence of the salt in the water somehow weakens the bond between the metal surface and the FeS precipitate. This could be caused by the $NaCl$ altering the particle size of the precipitating FeS , interfering with the formation of the FeS crystal lattice, or affecting the surface of the steel itself, preventing the FeS from adhering.

Whatever the cause, with no protective coating of FeS forming on the steel, the corrosion of the coupons continued at a higher rate over an extended period of time, as can be seen in Figure 3. With 1 percent $NaCl$, the FeS is washed off as it forms, and at the end of two weeks much more corrosion has taken place. With 4 percent $NaCl$, the weight loss at

the end of two weeks is more than with no salt, but is not so high as with 1 percent $NaCl$. The corrosion in 4 percent salt water lags behind that at 1 percent salt over the entire test period.

To study the effect of varying concentrations of salt more closely, an additional set of tests was made. Solutions containing varying concentrations of $NaCl$ in the water solution together with 540 ppm H_2S were prepared and run as before for a period of seven days. As before, no hard coating of FeS formed when salt was present. The results are plotted in Figure 4, showing the interesting result that a maximum in the corrosion rate occurs at a concentration of 1 percent $NaCl$.

The sharp rise in the curve of Figure 4 occurring between 0 and 1 percent $NaCl$ is caused by the loss of the protective FeS layer, as discussed above. At first it was thought that the decline in the extent of corrosion from 1 to 20 percent $NaCl$ might be due to a salting out effect, forcing the H_2S out of the water into the oil phase, thus reducing its effective concentration. Analysis of the solutions showed that such an effect does exist, but considering the relative insensitivity of the corrosion rate to H_2S concentration, it is not adequate to explain the observed results except, perhaps, in the range above 5 percent $NaCl$. The sharp drop in weight loss between 1 and 5 percent $NaCl$ must be ascribed to some other factor.

One possible explanation may be that $NaCl$ is, itself, a corrosion inhibitor. The observed behavior could be explained by Cl^- ion adsorbing on the metal surface. This adsorption would be expected to increase with increasing Cl^- ion concentration up to a certain point after which more Cl^- ion would have little effect. If the Cl^- ion were adsorbing, and a counterion layer of Na^+ ions forming, the H^+ ions would have to diffuse through this layer in order to reach the metal surface. Bearing in mind that the H^+ ion

is hydrated, steric factors could be important, not to mention the possibility of electrostatic repulsion from the Na^+ counter-ion layer. Adsorption of this type might also explain why the FeS precipitate was not bound so tightly to the metal. Another possibility is that it is not the Cl^- ion itself, but rather a complex of colloidal FeS precipitate and Cl^- ion which is adsorbing.

If any such adsorption is taking place, a corrosion inhibitor which depends on surface adsorption for its effect will have to compete for sites on the metal surface. For this reason, it would be well to set the NaCl concentration in a corrosion inhibitor test method at around 5 percent, where the phenomenon has had its

full effect. Any further increase in the NaCl content would only lower the effective H_2S concentration, and make more difficult the initial dissolving of the H_2S .

Conclusions

The initial rate of corrosion of steel coupons by oil-water- H_2S mixtures is dependent on the H_2S concentration. After an exposure time of approximately one day the rate becomes diffusion controlled and independent of H_2S concentration over a rather wide range. At high concentrations, the corrosion rate is actually lowered because of the formation of a denser, more crystalline FeS coating through which diffusion is slower.

Sodium chloride in concentrations up to 1 percent by weight increases the rate of corrosion, because it prevents the formation of a protective FeS coating on the steel. Above 1 percent NaCl , the corrosion rate falls off, possibly because the NaCl acts as a corrosion inhibitor and is adsorbed on the metal surface.

Acknowledgment

The authors express their appreciation to Dr. Norman Hackerman for his suggestions and advice.

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Any discussion of this article not published above
will appear in the December, 1959 issue

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Discussions on technical articles published in *Corrosion* will be accepted for review without invitation. Discussions must be constructive, accompanied by full substantiation of fact in the form of tables, graphs or other representative data and be submitted in three typewritten copies. In cases where illustrations are submitted, at least one copy of figures should be of a quality suitable for reproduction.

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An Analytical Procedure for Testing the Effectiveness Of Hydrogen Sulfide Corrosion Inhibitors*

By DONALD W. SHANNON⁽¹⁾ and JAMES E. BOGGS⁽²⁾

Introduction

A UNIFORM screening test for possible hydrogen sulfide corrosion inhibitors was proposed recently by a subcommittee of the National Association of Corrosion Engineers.¹ In this test, 100 ml of kerosene and 900 ml of a solution containing 500 ± 100 ppm H_2S and 5 percent NaCl are placed in a 1,000 ml conical flask. The inhibitor is then dissolved in the appropriate phase. A weighed steel coupon is held in the oil phase for exactly 10 seconds, then lowered into the aqueous phase, where it is left for seven days. After this period, the weight loss is determined and used as a measure of the extent of corrosion.

This test proposed to screen all materials, but upon examination it can be seen that it is inherently impossible to compare fairly a water-soluble substance with an oil-soluble substance. The amount of inhibitor added is based on the total volume of fluid, and, since there are 900 ml of water and 100 ml of kerosene, an oil-soluble inhibitor will have nine times the concentration of a water-soluble one. On the other hand, the coupon is exposed only 10 seconds to the oil phase, whereas it is in continuous contact with the water phase. Thus, the film life of an oil-soluble inhibitor is very important to the results, but does not matter for a water-soluble inhibitor. The test thus proposes to maintain in effect that a ten second exposure to any oil-soluble inhibitor at a certain concentration is equivalent to continuous exposure to a water-soluble inhibitor at one-ninth that concentration.

Since the proposed NACE method is designed as a screening test, a serious drawback exists in that a number of inhibitors which are effective under field conditions will show poorly or not at all on this testing procedure. In this category are such materials as:

(1) Any oil-soluble inhibitor depending for its effectiveness on surface adsorption that does not fully adsorb within 10 seconds.

(2) Any oil-soluble inhibitor depending for its effectiveness on surface adsorption which adsorbs in 10 seconds, but begins to desorb, or in any way lose its effectiveness, within 7 days when subjected to a corrosive water solution containing no more inhibitor.

(3) Any oil-soluble inhibitor which requires products of the corrosion reaction

to build a protective coating on the metal.

(4) Any inhibitor which requires the presence of oil in the protection mechanism.

This is a very formidable list of inhibitors to exclude from consideration.

Although this has not been checked experimentally, it would appear that the NACE test has a steadily decreasing corrosion rate, rather than a constant one. It has been shown in a previous paper² that the coating of FeS which builds up on the metal can slow the corrosion in this manner. Since the NACE test is a static one, an iron sulfide coating will form and remain on the coupons, while in actual field practice it might be continuously removed by the flowing liquid. Thus, a poor inhibitor will not show up as bad as it actually is because the corrosion products formed will protect the surface. This same objection could be raised against any static test method. Admittedly, a static test is simpler, and can be carried out more quickly with less equipment, but the effectiveness of a given substance as a corrosion inhibitor may be quite different in the two situations.

The NACE-proposed test has serious shortcomings which could result in dire consequences to the future of some commercial inhibitors should it become the nation-wide standard comparison test. The testing procedure presented in this paper has been set-up in such a manner as to avoid these difficulties.

Test Procedure

A description of materials and equipment used in the authors' test is given below.

Materials and Equipment.

Distilled water

Light mineral oil—USP grade (240 ml required for each test)

NaCl—analytical reagent grade

Hydrogen sulfide lecture bottle

Steel coupons—1 inch by 3 inch by 1/16th inch steel plate (1020 grade) with 1/8 inch hole drilled 1/4 inch from one end. All coupons to be cut from the same piece of sheet steel and to be prepared before use as described below.

Cylinder of nitrogen gas (with reduction valve)

0.1 N (0.05 molar) standard iodine solution

Soluble starch (indicator grade)

Inhibited HCl (6 N HCl containing "pickling" inhibitor)

Abstract

A new method is proposed as a standard screening test for possible hydrogen sulfide corrosion inhibitors. This is a dynamic, weight-loss method which attempts to control conditions sufficiently well that substances which offer promise of being effective inhibitors under field conditions can be selected for further study. The test is cheap and simple, so that it can be run economically by a relatively untrained technician on large numbers of materials.

The method is described and the reasons for choosing the particular conditions are explained. Then illustrations of the application of the procedure to a number of types of inhibiting substances are presented. 2.3.4

16 oz "French Square" screw cap bottles with metal caps
One gallon glass jug with two-hole rubber stopper
Bottle shaker—see descriptions below
Heavy-weight polyethylene film cut into 4 inch by 4 inch squares
3 mm glass rod
7 mm glass tubing
50 ml burette
125 ml conical flasks
25 ml pipette
0.1 ml pipette
0.2 ml pipette
0.5 ml pipette
No. 18 gauge copper wire
Rubber tubing
Y connector for rubber tubing
One pinch clamp
Three screw clamps
1 liter graduated cylinder
500 ml graduated cylinder
Analytical balance
Funnel

Construction of the Filling Apparatus.

Figure 1 gives a schematic diagram of the apparatus. This assembly will save much time in the handling of the solutions to be used.

Bottle Shaker.

Figure 2 shows a photograph of the device used for shaking the sample bottles. It is of simple construction, and is easily built. The sides are made from two 1 x 8 inch boards, and a 2 x 8 inch board is used for the center and ends. The bottles are held in place by a strip of wood fastened with three wing nuts.

A piece of rubber tubing is tacked to the underside of this strip. The shaker is driven by a 1/4 horsepower motor of 1460 rpm through a 20 to 1 reduction gear box, so that the rotation rate is 73 rpm. There is a metal-metal sleeve bearing at the motor end, and a metal-wood bearing at the other end.

This shaker worked very satisfactorily over a period of several months. By

* Submitted for publication January 28, 1958.

⁽¹⁾ General Electric Company, Hanford Atomic Products Operation, Richland, Washington.

⁽²⁾ Chemistry Department, The University of Texas, Austin, Texas.

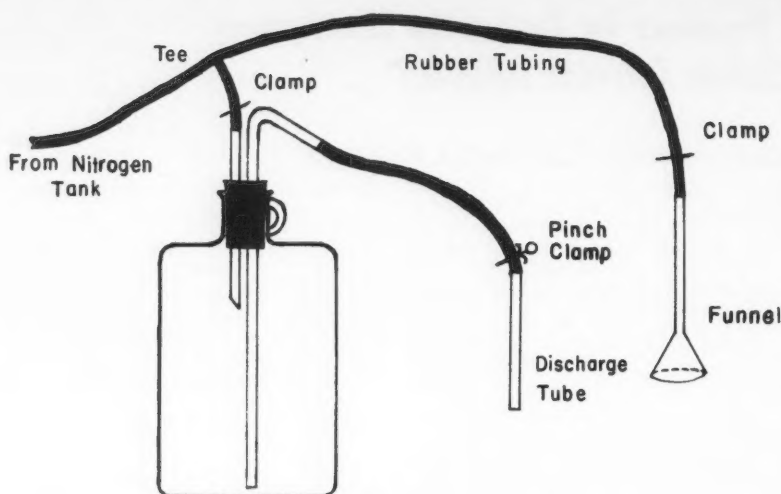


Figure 1—Apparatus for filling sample bottles.

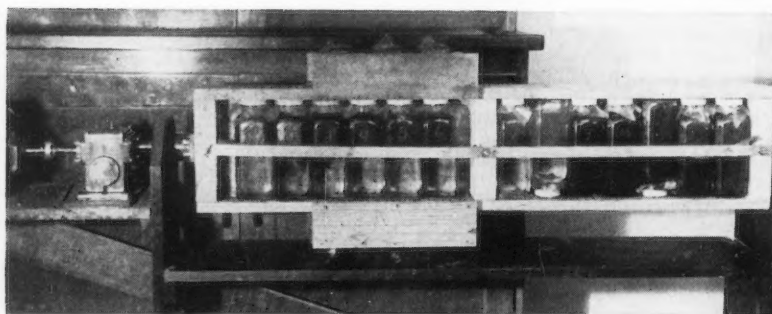


Figure 2—Apparatus for rotating sample bottles.

using four sides instead of two, a shaker to hold 64 bottles could be made.

Preparation of Coupons.

The coupons should be mechanically sanded with fine (No. 2) emery paper to give a uniform new metal surface. High polishing to a mirror-like surface is unnecessary. All grease, emery dust, fingerprints, etc., should be removed with soap, water, and fine steel wool. As soon as this washing is begun, care must be taken not to touch the metal surface with the fingers. The coupons should be handled by means of a glass hook or forceps. The clean coupons should be rinsed to remove any soap, dipped in distilled water, and then dried by washing with acetone. Care should be taken not to let any soap dry on the surface before rinsing.

Clean coupons should be weighed to the nearest 0.1 mg and the weights recorded. As each coupon is weighed, it should be wrapped in paper or placed in an envelope, and its number written on the outside.

The coupons should be stored in a desiccator until used. The new metal surface rusts very rapidly if left exposed.

Preparation of Solution

Place three liters of distilled water in the glass jug. This gives enough solution for ten tests. Bubble nitrogen gas

vigorously through the distilled water to remove any dissolved oxygen. If a large amount of oxygen is dissolved, it will help to pull a vacuum over the water first. The length of the wash depends on the amount of oxygen present, but about 15 minutes is usually sufficient. If when the H_2S is introduced, the solution turns even slightly cloudy, all the oxygen was not removed, and the solution should be discarded.

Add 155 gm of NaCl, weighed to the nearest gram, to the water, forming a 5 percent solution. The bubbling of the nitrogen can be used for stirring to dissolve it.

Wire the stopper on the jug and connect the H_2S cylinder to the discharge tube. The required concentration of H_2S is 1500 ± 50 ppm. (CAUTION: H_2S is dangerous. Take precautions to avoid undue exposure. Work in a hood.) Place about five pounds pressure of H_2S in the jug. Shake the jug vigorously, then bring the H_2S pressure back up and repeat the shaking. In this manner the required concentration can be introduced in a few minutes without exposure to H_2S . A little experience is necessary to know how many shakes are required, but try about ten cycles of shaking and bringing the pressure back up before checking the concentration. After practice, the amount of gas to be put in can be estimated closely.

Open the jug in a hood and withdraw a 25 ml sample. (Do not pipette by mouth!) Titrate the sample in a 125 ml conical flask with standard 0.1 N iodine (0.05 M) using freshly prepared starch solution as indicator. Add about 90 percent of the iodine before shaking, in order to reduce the losses: The last traces of H_2S react rather slowly. The reaction is:



Thus, one equivalent of iodine equals one-half mole of H_2S . Since ppm is mg/liter, the ppm of H_2S may be calculated by:

ppm. H_2S =

$$\frac{\text{ml iodine} \times \text{norm. iodine} \times 17,040}{\text{ml of sample}}$$

Adjust the concentration to 1500 ± 50 ppm. If too much H_2S has been put in, it can be removed by a quick wash with nitrogen.

Mounting of Coupons

Ten weighed coupons are required for each inhibitor to be run. Place each coupon in the bottom of a bottle, diagonally across the square, hole up. A piece of 3 mm glass rod is then cut to fit diagonally from the bottom of the bottle to the top, passing through the hole. These rods should be cut about $\frac{1}{8}$ inch short to allow for variations in the sizes of the bottles. If they are too long, they will break when the caps are screwed on. Fire-polish the ends so that they will not cut the plastic film. These rods may be used over and over. Mark each bottle with the coupon number.

Addition of Oil

Add 240 ml of light mineral oil to each bottle. Allow a few minutes for gas bubbles to escape.

Addition of Inhibitor

Use duplicate samples at 10, 25, 50, and 100 ppm of inhibitor (based on 480 ml total fluid) and two blanks. If the inhibitor is water soluble, place the drop of inhibitor on the bottom of the bottle under the oil layer. Do not shake. When the water is added in the next step, the inhibitor will be dispersed.

Addition of Water

Wire the stopper of the jug in place and put in about five pounds pressure of nitrogen. Place the discharge tube beneath the surface of the oil in a sample bottle, open the pinch clamp, and introduce 240 ml of water. This is measured by finding out the position on the bottle that corresponds to 480 ml (allow for the volume of the discharge tube) and adding water until the oil layer reaches this mark. All the bottles are filled in this manner, so that no oxygen comes in contact with the water. Proceed immediately to the next step, since the H_2S will start diffusing into the oil and out of the bottle if left uncapped.

Addition of Nitrogen Gas

Start a vigorous discharge of nitrogen gas out of an inverted funnel. Place the

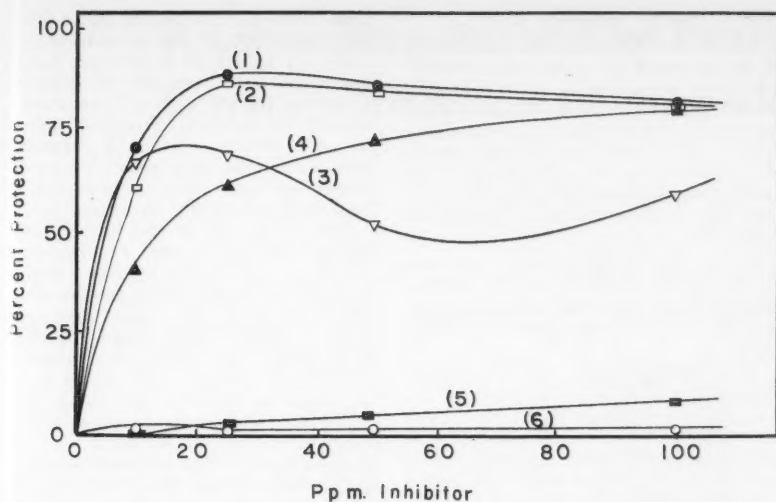


Figure 3—Percent protection by various inhibitors as a function of inhibitor concentration. Code to numbers is as follows: (1) Diethylenetriamine, (2) Triethylenetetramine, (3) Duomeen 12D, (4) 2-Mercaptobenzothiazole, (5) Sodium pyrophosphate, (6) Benzoic acid.

test bottle under this funnel for 10 to 15 seconds to sweep out any air in the bottle. Keeping the bottle under the nitrogen jet, slip a 4 inch by 4 inch piece of polyethylene film over the top. Holding the film taut with the thumb and forefinger of the left hand, remove the bottle and screw on the cap. Take great care that there are no wrinkles in the plastic along the top rim of the bottle. Finally tighten the cap. In a similar manner seal all the bottles.

Duration of Test

Place the bottles on the shaker and begin rotation. This rotation must continue 24 hours a day for seven days. After this period remove the bottles from the shaker and open in a hood. The sound of escaping gas should be heard as each is unsealed if much corrosion has occurred.

Cleaning of Coupons

Wipe off all oil and loose material with a paper towel. Dip each coupon in inhibited HCl solution for about ten seconds to loosen any remaining scale. Then wash each coupon with soap and water using fine steel wool. Rinse with water and dry with a clean towel. Be careful not to touch with the fingers. Dip in acetone and, when dry, reweigh to 0.1 mg.

Calculation of Results

Calculate percent protection:

percent protection = 100 —

$$\frac{\text{wt loss of inhibited coupon} \times 100}{\text{wt loss of blank coupon}}$$

Calculated in this way:

Perfect protection = 100

No protection = 0

Catalytic effect = negative result

Plot percent protection vs ppm inhibitor added.

Explanation of Test Method

In general, the choice of conditions for the test method has been based on two fundamental considerations. First, the procedure must be run under conditions such that, to the best of present knowledge, it will pick out substances, regardless of their nature, that are likely to be useful corrosion inhibitors in the field. These promising materials can then be investigated more completely. Secondly, the test procedure must be sufficiently cheap and simple that it can be run economically on large numbers of substances by a relatively untrained technician.

Test Container. In the past, various pieces of laboratory glassware—flasks, side arm test tubes, etc.—have been used for corrosion test containers. All such glassware is expensive. If the containers are used only once, they can represent a sizeable expense over a period of time. If they are reused, the costs involved in washing must be considered. Besides the financial question, there is the fact that, since inhibitors are surface-active agents, there is always the chance that some inhibitor adsorbed on the glass will carry over from one test to another. Very small concentrations of inhibitors are used, so just a little contamination would affect the results. The bottles chosen are readily available anywhere, convenient to use, and very cheap so that they may be discarded after use.

The method of sealing has been shown² to be effective and not to introduce any substance that will interfere with the test. A sealed, all-glass container, such as that suggested by Caldwell and Lytle³ would have certain obvious advantages. However such tubes are expensive to use when hundreds of tests are to be run, and a certain amount of glass-blowing skill is needed for proper sealing. The method the authors have adopted is simple and requires no special skill. Both the metal and plastic

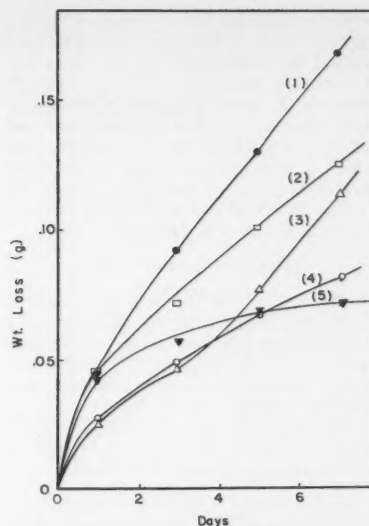


Figure 4—Extent of corrosion in the presence of various inhibitors as a function of time. Code to numbers is as follows: (1) No inhibitor, (2) Carbon monoxide, (3) Potassium dichromate, (4) 2-Mercaptobenzothiazole, (5) Duomeen 12D.

caps were tried on bottles with the metal caps proving to be more foolproof. The plastic caps could be broken too easily in tightening.

Solutions. Some inhibitors are oil-soluble, others are water-soluble or dispersible, and some do not fit either classification, but are somewhat soluble in both phases. If all these materials are to be compared with one another as to their inhibiting properties, the test system must be such that both the oil phase and the water phase come in contact with the metal surface, so that regardless of which phase the inhibitor is in, it will reach the metal. Also, some inhibitors require a time interval of contact before they become effective; for this reason this mutual contact of water and oil to the steel must be maintained over the length of the test.

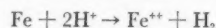
The corrosion process of a static system is certainly different from the process in a dynamic situation, if for no other reason than that the corrosion products and the inhibitor adsorbed on them may be washed off in the dynamic one. A dynamic test was chosen because, in addition to the necessity of bringing both phases in contact with the steel, flowing conditions more realistically simulate the actual conditions under which inhibitors are used.

Equal volumes of oil and water were used, so that if equal amounts of oil-soluble and water-soluble materials were added, their concentrations would be the same.

It has been the practice in some tests to use "sour" crude oil as the oil phase. This seems highly undesirable for the reason that such oil is completely irreproducible. The composition of crude oil is different from well to well, and changes from day to day in the same well. Also, some oils are known to contain materials which are natural cor-

rosion inhibitors. Other purified hydrocarbons, such as kerosene, could be used instead of mineral oil, but on the whole it was felt that the USP grade light mineral oil would present a more uniform composition and freedom from impurities regardless of the source from which it was obtained and would have similar physical properties to crude oil.

The 30 ml of gas is essential. The reaction at the steel surface is:



If the bottles were completely full of liquid, the pressure of the hydrogen produced would either cause the cap to leak or would break the bottle. Nitrogen was chosen because it is inert, cheap, and readily available.

The concentrations of H_2S and NaCl were chosen for the test on the basis of experiments described earlier.²

Steel Coupon. The steel coupon was cut to the same size as a standard microscope slide to facilitate examination. Mild (1020) steel was chosen since it is so widely used in actual construction. The inhibition of corrosion on other metals or alloys could be studied in a similar manner. The fact that pieces of steel with different past histories will corrode differently poses a major problem for any standard test. Since the test described is intended as a screening procedure to compare the relative effectiveness of various inhibitors, the problem can be most nearly overcome by cutting all of the coupons used from the same piece of steel.

The cleaning procedure used to remove FeS from the coupons after a corrosion test and before weighing has been shown to be reproducible and effective. The loss in weight of blank coupons subjected to this treatment was always less than 0.0005 gm. No difficulty was encountered in reusing the coupons for additional tests if they were polished on the emery wheel and carried through the standard initial cleaning procedure.

Sample Inhibitor Tests

As an illustration of the effectiveness of the proposed test in screening possible inhibitors, six materials of widely different types were chosen to be ranked as to their effectiveness as hydrogen sulfide corrosion inhibitors. All were run in the standard way for a period of 7 days, using varying concentrations of inhibitors. The results obtained are plotted in Figure 3.

It can be seen that benzoic acid had no detachable inhibiting action. Sodium pyrophosphate had some effect, but not enough to be of any practical significance.

The inhibitor activity of 2-mercaptobenzothiazole continued to rise with increasing concentration throughout the test range. Diethylenetriamine and triethylenetetramine were the most effective of the corrosion inhibitors tested (although much better ones are known), and appear to be equally effective. The shape of the curve for Duomeen 12D is interesting and can be correlated with observations of the coupons during the course of the run. A heavy coating of oil was found to adhere to the coupons at low inhibitor concentrations. Duomeen 12D is an excellent emulsifying agent, and at the higher concentrations strongly emulsified the test solutions, leaving a much lighter oil coating on the coupons. Thus at higher concentrations Duomeen 12D decreases the thickness of the protective oil coating and is less effective as a corrosion inhibitor than at low concentrations. An effect of this nature would not have been discovered using the NACE test procedure.

The test method outlined also can be used to study the variation of corrosion inhibition efficiency with time. Four inhibitor materials (carbon monoxide, potassium dichromate, 2-mercaptobenzothiazole, and Duomeen 12D) were chosen to be added in excess to the test system. In the carbon monoxide tests, 25 ml of CO gas was placed in the gas

phase above the two liquids. Since potassium dichromate reacts with H_2S , it could not be added to the test bottles (nor could it economically be added to the crude oil line in field operations). Instead, the coupons were first dipped into a 1 percent solution of potassium dichromate, and then placed in an inhibitor-free test system. The results of these tests are plotted in Figure 4.

From Figure 4, it can be seen that neither carbon monoxide nor Duomeen 12D show any inhibiting effect during the first day, while the protective coating is being formed. After this time, carbon monoxide has a slight inhibiting effect. Since the curve for Duomeen 12D flattens out, it would appear to be an excellent inhibitor over long time periods. The 2-mercaptobenzothiazole acts quickly, but the corrosion then appears to proceed in a continuous, though considerably slowed, manner. Potassium dichromate protects well at first, but after about three days in the absence of a fresh supply of inhibitor, the protection disappears and the corrosion rate (as shown by the slope) is the same as with no inhibitor present.

It can be seen from the data presented for the sample inhibitor tests that in many cases the effectiveness of an inhibitor depends on the conditions of contact between the inhibitor, the metal, and the two-phase corrosive medium. It is for this reason that this proposed test was developed. It meets the conditions of simplicity and economy, and also comes closer than many tests to duplicating actual field conditions. The test is a dynamic one, with both oil and water phases flowing over the metal, and the inhibitor is given equal chance to protect the surface regardless of its relative solubility in the two phases.

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Any discussion of this article not published above will appear in the December, 1959 issue

TECHNICAL PAPERS ON CORROSION WELCOMED

Authors of technical papers on corrosion are invited to submit them for review without invitation to the Editor of *Corrosion*. Write for "Guide for the Preparation and Presentation of Papers" sent free on request to prospective authors.

A Laboratory Method for Evaluating Corrosion Inhibitors for Secondary Recovery*

By T. R. NEWMAN

Introduction

ONE OF the more serious problems that confronts the water flood operator is corrosion. Unless this problem is controlled it can result in a premature termination of the flooding operation. This has occurred in many cases and as a consequence the recovery of oil has been considerably less than anticipated.

There are many different ways in which the effects of corrosion are manifested. The most obvious are the perforation of carrying lines and the reduced life of equipment. In addition, however, corrosion products may react with some of the constituents of the brines used for flooding, forming insoluble products which are filtered out by the formation face. This results in lower injection rates and necessitates higher injection pressures. Eventually it can lead to complete plugging of the formation.

The control of corrosion can be accomplished either by the use of equipment that will not react with the brine, or by rendering the fluid less corrosive. Plastic or cement lined equipment is used in some instances, but where expected flood life is low it is generally more economical to treat the brine chemically to reduce the corrosivity. Corrosion inhibitors have found wide acceptance for this purpose. Unfortunately, there is no known corrosion inhibitor that is effective in every water. This variation in inhibitor performance has necessitated the evaluation of inhibitors under the environmental conditions for which it is being considered. A trial in a pilot flood is one of the best ways of checking inhibitors, but this is often too costly and time consuming and therefore, a reliable laboratory test method would have considerable value. Such a method has been developed and is described in detail in this paper.

Field Conditions

A secondary recovery system, in its simplest form, consists of an injection well, a producing well, and a water source. These points are generally connected by carrying lines and usually include reservoirs, tanks, filters, pumps, etc.

There are two basic types of systems for water flooding: open and closed. In an open system the injection brine comes in contact with the atmosphere. In a closed system, air is excluded. Stabilization of calcium, barium and other metallic ions to prevent their deposition is seldom a problem in closed systems, but it is often necessary to add polyphos-

About
the
Author



T. R. NEWMAN is Research Chemist, Physical Chemistry Laboratories, National Aluminate Corporation of Chicago. Mr. Newman received a BS in chemistry from the University of Chicago in 1949. He joined the National Aluminate Corporation in 1949 and has been with them since. Mr. Newman's fields of research have included coagulation, corrosion and analytical techniques. For the past several years his work has been concerned with corrosion in aqueous systems.

phates for this purpose in open systems. Metals such as iron or manganese in solution are undesirable because they can form insoluble particles. They can be controlled either by the use of certain organic acids or chelating agents.

The most common metal used in water flooding equipment is mild steel. Copper alloys or light metals are not resistant enough to the brines used. Therefore, corrosion in water flooding is almost exclusively that of ferrous metals.

Environmental conditions may vary considerably from one waterflooding area to another. Their effect on the nature and amount of corrosion is considerable, and any laboratory test for waterflood corrosion studies must give due consideration to the conditions existing in the particular area under study.

The range of temperatures generally encountered is from 80 F to 120 F. Exposed surface lines can reach the upper limit but seldom are higher. Of course, the subsurface system will have higher temperatures, but this constitutes only a small part of the total carrying lines.

The movement of fluids in the lines is moderate. There are few stagnant areas. The flow will be in the range of several feet per second.

The waters used for sub-surface injection are generally saline. There are some areas where fresh waters are injected, but these are the exception rather than the rule. Injection of fresh water can lead to swelling of some clay constituents of the formation, and for this reason this procedure ordinarily is not followed. In addition, fresh water is seldom available in the quantities needed for water flood-

Abstract

A new test method is presented for evaluating corrosion inhibitors for secondary recovery systems. The test is characterized by its relatively high ratio of volume of water to surface area of metal exposed.

Variables studied included dissolved oxygen, H_2S , dissolved solids and pH. Corrosivity of a brine was found to be almost directly related to the dissolved oxygen level. It was found also that a high H_2S system was not necessarily more corrosive than one having a low level of H_2S , but that the attack became more localized. The higher the H_2S level, the higher the inhibitor level required to obtain an equivalent level of protection. The greatest effects of variations in dissolved solids are indirect. The solubilities of dissolved gases and organic inhibitors are inversely proportional to the level of dissolved solids. The variation of pH in the range from 5 to 8 did not affect the performance of a typical inhibitor in a system without H_2S . When H_2S was present, the protection with this inhibitor dropped off slightly as the pH was lowered to 5.

This test method was used to evaluate a number of commercial water flood inhibitors. Experimental inhibitors were tested until one was found that exceeded the performance of those now being used. This one was tested in several floods and the excellent performance, predicted from the laboratory results, was confirmed. 5.8.1

ing. The salinity of the waters can range as high as 25 percent, with some instances of higher brines reported. The dissolved gases in these brines also vary widely. Hydrogen sulfide, carbon dioxide and oxygen are the ones of greatest concern in water flooding. Hydrogen sulfide varies from trace amounts up to around 600 mg/liter in extremely sour areas. Carbon dioxide also varies over a similar range. Dissolved oxygen ranges from practically zero up to approximately 8 mg/liter in brines.

Generally the pH values of injection brines are not too far removed from the neutral point, but there are waters where the pH is quite low due to the presence of dissolved acidic gases. This occurs mostly in closed systems. Open systems generally have waters with a pH near neutral.

Experimental

Apparatus

The test developed for studying corrosion inhibitors for water flooding is essentially a recirculating system. The apparatus as shown in Figure 1 consists of a 5 gallon reservoir, a centrifugal pump, and a plastic cell for holding the coupons. The plastic cell was formed from a 1-inch lucite core and two 1/4 inch lucite cover plates. The channels were cut in the core, and the holes drilled and tapped. The cut surfaces were finished off, and then the cover plates were glued on under pressure. The dimensions of the finished cell are 6 1/2 x 8 1/2 inches for the 3 coupon unit. Cells measuring 8 1/2 x 13 inches have been made for holding 7

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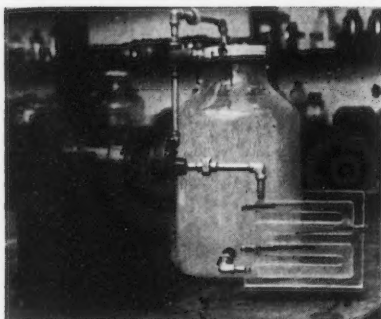


Figure 1—Apparatus for holding coupons.

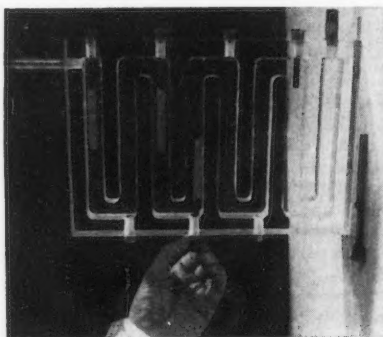


Figure 2—Block for coupon testing.

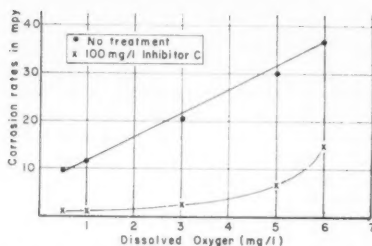


Figure 3—Effect of dissolved oxygen in 3 percent brine with 10 mg/liter hydrogen sulfide.

coupons. The channels are $\frac{3}{4}$ of an inch high in the 1 inch core. Figure 2 is a photograph of this larger block.

The test coupons are held in the channels by coupon holders. They are made from $\frac{1}{4}$ inch stainless steel pipe plugs that have been drilled and tapped on the threaded end. The hole accommodates a $\frac{1}{4}$ inch hard rubber rod. The free end of the rod is prepared to hold the coupon with a $\frac{5}{32} \times \frac{1}{4}$ nylon bolt. The coupons are bolted to the holder and then screwed in place in the plastic block.

The coupons used in this work were $\frac{3}{4} \times 3$ inches, and were of 16 gauge SAE 1010 mild steel. They have a $\frac{5}{16}$ inch hole drilled at one end to accommodate the nylon bolt. The coupons are sand blasted before use. They are also rinsed in toluene and acetone prior to use. At the conclusion of the test they are rinsed free of loose deposit in running tap water, then acid cleaned in muriatic acid inhibited with formaldehyde. After the 30 second acid dip they are neutralized in a soda ash solution, washed in running water, and then dipped

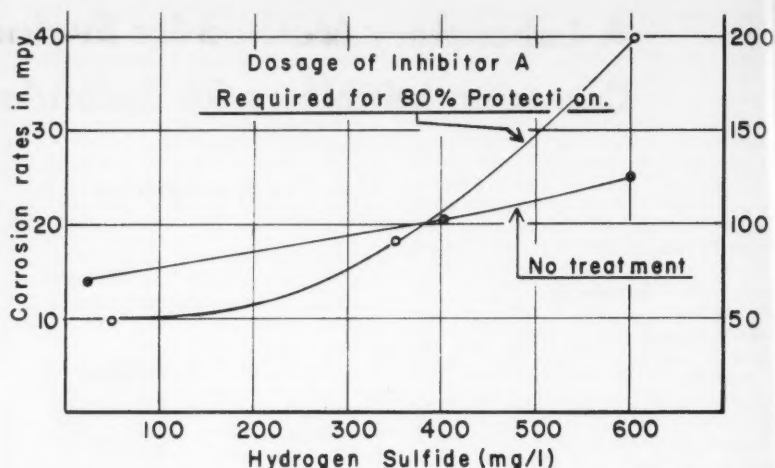


Figure 4—Corrosivity of 10 percent brine containing hydrogen sulfide and less than 1 mg/liter of dissolved oxygen.

in acetone and dried. The coupons can then be weighed. The cleaning loss is determined by repeating the procedure. The corrosion rates are then calculated.

Corrosometer¹ probes also can be used in the test block. These devices have the obvious advantages of enabling one to keep a continuous check on corrosion rates, and also find use in studying film persistence. Probes were generally used in tests of longer duration than 1 day, because of the ease with which repetitive readings could be made, in contrast to the limited number possible with coupons.

The three component parts of this apparatus are interconnected with $\frac{1}{4}$ inch No. 304 stainless steel pipe. The flow through the system is from the reservoir to the bottom of the plastic block and out the top. Then the water enters the pump and proceeds through the gate valve and back to the reservoir. The test cell is on the suction side of the pump to minimize leakage. The pump is an Eastern Industries Model D-11 stainless steel centrifugal pump. The flow rate is set at $5\frac{1}{2}$ liters per minute.

Test Conditions

Some of the initial work was done on a synthetic 3 percent brine. This was used because it was typical of water in the initial survey of some areas where flooding was being done. Since that time, additional surveys of water flood systems have shown that brines higher in dissolved solids are more representative of areas where much of the flooding is taking place. A representative 10 percent brine was synthesized for this work. Typical analyses of these two brines are given in Table 1.

The mineral contents of the brines are relatively simple to duplicate. A more difficult task is to provide a brine having a dissolved gas content similar to that occurring naturally. With this test, the best means for accomplishing this is to sweep the reservoir with a blend of the gases in ratios that will give the desired dissolved gas content in solution. In the recirculating test this was done by

inserting a perforated plastic cylinder in the reservoir and introducing the gas into its center through a gas diffusion tube. The gases were metered using Fisher and Porter Flowraters. The composition of the test brine was checked using standard APHA methods when possible. The dissolved oxygen level was determined with the Dorber^{*} rotating platinum electrode meter.

In most areas of corrosion studies, numerous test methods have been published. The lack of published methods in the area of water flooding may be due to the difficulties inherent in the duplication of actual operating conditions. The mineral content of the water is generally easy to duplicate in the laboratory, but the dissolved gas content can be very difficult to reproduce. The relationship between the various dissolved gases must be maintained in order to simulate the corrosivity of the system. Water flood systems are essentially "once-through," and therefore the volume of water per unit of surface area of metal exposed is quite high. Also, because of the large volumes of fluids involved, only low dosage levels of inhibitors are economically practical.

The temperature of the brine during the test was 105 ± 5 . The tests were started with the brine at that temperature. This temperature in the test apparatus was maintained without the application of external heat. This was possible due to the introduction of heat by friction and the shearing action in the impeller section of the pump.

Most water flood inhibitors are surface active. Because of the tendency of such materials to attach themselves to any solid surface, it was felt that a high ratio of water to metal surface was desirable. A continuous replenishment of the protective film must also be provided. These two objectives were accomplished by designing a test having a large reservoir and a test cell that would hold the test panels in the flow of the fluid. The ratio of water volume to metal surface area in this test is about 3 liters per square

^{*} Dorber Company, Chicago, Illinois.

inch, whereas in other typical laboratory tests the ratio generally is about 100 to 200 ml per square inch.

The use of a greater volume of test water than found commonly in laboratory tests also has other advantages. The composition of the test fluid will change less during the course of the test. In some batch type aqueous tests, for example, the pH may increase as much as one unit in a 24 hour period. Also to be reckoned with is the consumption of inhibitor. In many batch type tests the consumption of a surface active inhibitor can be in excess of 50 percent in the first 24 hours when low concentrations are used. In this recirculating test, the consumption of inhibitor does not exceed 25 percent.

Procedure

To prepare for setting up a test, the first step is to make the test water by addition of the appropriate chemicals to tap water in the reservoir. The water temperature is adjusted to $105\text{ F} \pm 5$, and maintained there throughout the test. Next the inhibitor is added to the water. Then the test block is isolated from the reservoir with either a valve or a cork stopper placed into the pipe at the bottom of the reservoir. In the low sulfide tests (1-20 mg/liter H_2S) coupons are then inserted, the system assembled, and the pump started. H_2S is then introduced.

In high H_2S (>200 mg/liter) tests the reservoir is swept with a blend of N_2 and H_2S prior to the insertion of the coupons until equilibrium is reached (1-2 hours). Then the coupons are put in place and circulation started. The test is generally allowed to run for 24 hours. When it is desired to observe pitting an exposure period of at least one week is necessary.

To calculate the data, corrosion rates for an untreated system are needed. This figure is expressed as zero protection. The rates with inhibitors are converted into percent protection from this base. The test coupons were always examined for the type of attack. Pitting was evaluated visually.

The test period can be of almost any desired length of time. Corrosion studies in the field generally require exposures of two weeks to a month. For laboratory evaluations this would be too long, and so the test was designed to yield results in 24 hours. The validity of the one day results was confirmed by checking against one week tests. Excellent correlation was obtained between the tests run for different exposure periods.

Results

Reproducibility

One of the primary requirements for a laboratory test is that the results can be reproduced. In this particular testing procedure there are two different areas where reproducibility is needed: (a) the coupons within a given test block should correlate with each other and (b) results from one run to the next should be reproducible. The work shown in Table 2 which was carried out in 10 percent brine with the low H_2S level and less than 1 mg/liter dissolved oxygen was excellent. The duration of these tests and of all the other tests described in this paper unless otherwise specified was 24 hours.

The results with commercial inhibitors in this test also correlated well with the available field data. The same order of effectiveness was obtained for these materials both in the laboratory and in several applications in the field.

During the course of this work many different inhibitors were studied. Data on three typical materials are given in this paper. They are represented by the letters A, B, and C. They are all high molecular weight nitrogenous organic formulations. Inhibitor A is an oil based formulation, while B and C are water based. Inhibitor A was developed with the aid of the test described in this paper. Inhibitors B and C are typical commercially available inhibitors.

The Effect of Dissolved Oxygen

The effect of dissolved oxygen on corrosion was essentially linear in the range studied (the corrosion rates for the blanks increased as the dissolved oxygen level increased). These results are shown in Figure 3 for work done in the 3 percent brine with 10 mg/liter H_2S . Corrosion inhibition by a commercial water flood inhibitor (Inhibitor C) was fairly good until the dissolved oxygen reached about 4 mg/liter in this test. As the dissolved oxygen level was increased above this point, the protection dropped off. With 100 mg/liter of Inhibitor C the protection below 1 mg/liter of dissolved oxygen was as high as 90 percent, but as the oxygen was raised to 5 mg/liter, only 55 percent protection was obtained. At dissolved oxygen levels near saturation, very little protection was obtained with available inhibitors at 100 mg/liter or less.

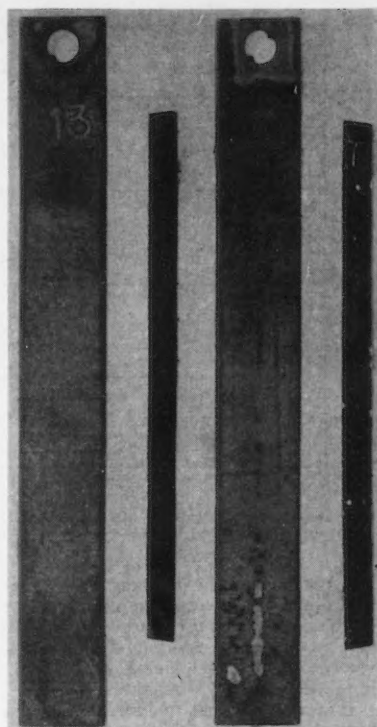


Figure 5—Probe elements from Figure 4 tests.

Effect of H_2S

The corrosivity of the 10 percent brine containing less than 1 mg/liter of dissolved oxygen increased with increasing levels of H_2S , but not as sharply as might be expected. The results are given in Figure 4. A significant observation of this effect was that the treatment requirements with Inhibitor A went up as the H_2S level was increased. In other words, to obtain the same degree of protection in two tests of low and high sulfide levels, the high sulfide test would require about five times more inhibitor. Also, in the high H_2S system the attack was localized and severe pitting occurred. In a one week test, perforation of the Corrosometer strip probe occurred in several places. Figure 5 shows the probe element from these tests.

Effect of Dissolved Solids

The early work with the 3 percent brine was designed to aid in understand-

TABLE 1—Typical Analyses of Synthetic Test Waters

	Low Brine (mg/liter)	High Brine (mg/liter)
Total Dissolved Solids.....	38,000	104,000
Total Hardness (as CaCO_3).....	6,800	1,895
Calcium Hardness (as CaCO_3).....	4,400	1,350
Magnesium Hardness (as CaCO_3).....	2,400	545
"P" Alkalinity (as CaCO_3).....	0	0
Total Alkalinity** (as CaCO_3).....	50	90
Chloride as NaCl	36,200	102,000
Sulfate as Na_2SO_4	19	40
Ba & Sr as combined sulfates.....	310	0
pH.....	7.50	7.2

* Alkalinity to phenolphthalein indicator.

** Alkalinity to methyl orange indicator.

TABLE 2—Correlation and Reproducibility of Tests

	Reproducibility of Test ^a		Average mpy
No Treatment..	(1) 13.6—14.9—13.2 ^b (2) 13.6—15.0—13.8		13.9 14.1
	Results with Inhibitor A (Percent Protection)		
	10 mg/l	25 mg/l	50 mg/l
Test No. 1.....	48, 49, 54 ^b	66, 64, 70	76, 75, 79
Average.....	50%	66%	77%
Test No. 2.....	48, 46, 41	70, 64, 61	72, 77, 73
Average.....	45%	65%	74%

* Ten percent brine with low H_2S level and under 1 mg/liter dissolved oxygen.

^b The groups of 3 figures are from the three coupons in one test cell.

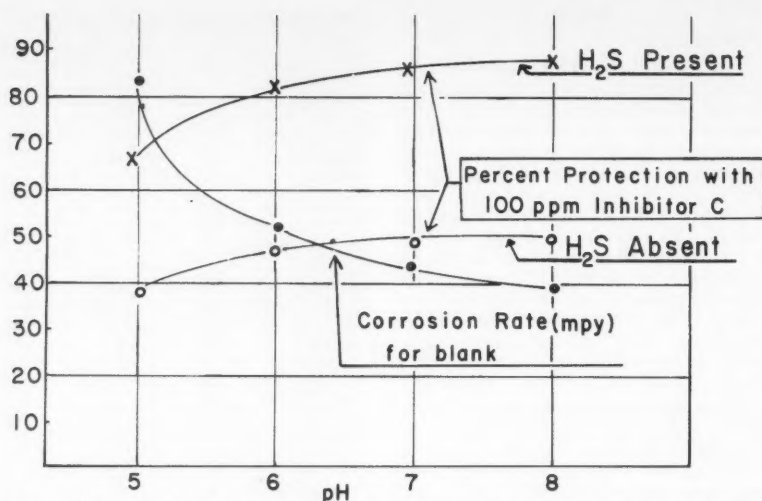


Figure 6—Relationship between pH and corrosivity in 3 percent brine containing 10 mg/liter hydrogen sulfide and 4 to 5 mg/liter of dissolved oxygen.

ing more about the factors and mechanisms involved in corrosion and corrosion inhibition in secondary recovery. The 10 percent brine was used in the work involved in the development of new and improved corrosion inhibitors.

It was found that the corrosivity of a brine having a low H_2S level and dissolved oxygen below 1 mg/liter did not appear to have a direct relationship to the dissolved solids content. Corrosion rates for blank tests with 3 percent and 10 percent brines (all other factors being constant) were 12 mpy and 14 mpy. Generally other factors such as dissolved gases and pH affected the corrosion rates to a greater extent.

Substances dissolved in water increase the conductivity, but an increase in dissolved solids in a water already containing a high proportion of solids in solution, and therefore having a high conductivity, seemed to have little effect on the rate of corrosion.

The aggressiveness of chlorides is well known, and most injection waters are high in chlorides. But again in discussing the effect of chlorides in brines, the comparison is not between a water free of chlorides and one high in chlorides. When the corrosivity of a brine containing 3 percent NaCl is compared with one containing 10 percent the effect is small. The change in the solubility of gases, and specifically the decrease in oxygen solubility, tends to offset the increase in corrosivity due to the presence of increased numbers of chloride ions.

Effect of pH

Studies on the effect of pH were carried out by adjusting the pH with small amounts of HCl and NaOH. It was found that as the pH increased from 5 to 8 the corrosivity decreased by approximately half. Corrosion inhibition with Inhibitor C, in the case of 3 percent brine containing low H_2S and from 4 to 5 mg/liter of dissolved oxygen, was somewhat poorer at the low pH level than at pH 8. These results are shown in Figure 6. Corrosion inhibition with Inhibitor C in the same brine without H_2S ,

was affected less by pH change from 5 to 8. The protection obtained was less in this case over the pH range studied. It is recognized that lower levels of pH are found in some closed systems, but most injection brines will fall in the range studied.

Results with Inhibitors

The dosage levels of corrosion inhibitors in laboratory tests are characteristically higher than those required in practice. This is partly because of the time element. In practice a longer period is generally allowed for the formation of the inhibitor film. The value of a laboratory test is diminished if the exposure period required is too long. Laboratory tests are usually made more severe for this reason, and as a consequence, higher treatment levels than those used in practice are generally necessary. The test presented in this paper has succeeded in decreasing the discrepancy between laboratory and field dosage levels, but a difference still exists. For example Inhibitor A which requires about 25 mg/liter for an appreciable degree of protection in the laboratory test, is generally used at about 10 mg/liter in the field.

Inhibitors were studied at levels ranging from 5 mg/liter to 100 mg/liter. It was found that many materials gave good protection at 50 or 100 mg/liter, but at lower dosages, the differences between inhibitors became more apparent. The economics of water flooding are such that low levels of treatment must be used. Therefore, a comparison of inhibitors at lower dosages is believed to be more significant than that at higher concentrations.

An important characteristic of good inhibitor performance was found to be a relatively gradual slope of the curve for percent protection vs treatment level. About 50 percent protection was found at 10 mg/liter and 80 percent at 50 mg/liter. Many inferior inhibitors (based on field results) gave as high as 90 percent protection at 50 mg/liter, but showed no effect at 10 mg/liter. Data

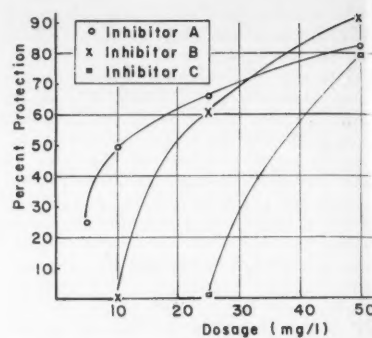


Figure 7—Dosage vs percent protection for inhibitors in 10 percent brine containing 10 mg/liter hydrogen sulfide and less than 1 mg/liter dissolved oxygen.

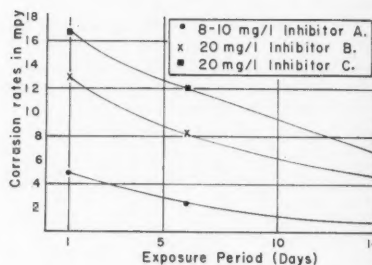


Figure 8—Results of field tests with three inhibitors.

on three typical inhibitors are given in Figure 7.

Field reports on the inhibitors studied have substantiated the results obtained in this laboratory test. Inhibitor A has been found to give superior protection over Inhibitors B and C under several different sets of conditions. In a particularly corrosive system having an H_2S level of about 600 mg/liter, Inhibitor A has virtually stopped the pitting attack and is giving excellent protection. In several other applications, Inhibitor A has given better protection at 8 to 10 mg/liter than two other commercial inhibitors have shown at 20 mg/liter. Figure 8 shows such results for a flood having a brine content of 10 percent, low H_2S , and < 1 mg/liter of dissolved oxygen.

Summary

A new test method has been developed that is particularly suitable for evaluating corrosion inhibitors for water flooding. A corrosion inhibitor developed with this method has been tested in several field applications and good correlation has been found to exist with the lab results.

Acknowledgment

The author wishes to express his appreciation to J. I. Bregman, Jerome Green, L. L. Bott and D. C. Olen for their advice and guidance during the course of this work.

Reference

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Wash Primer Development and Characteristics*

By L. R. WHITING

Introduction

PURPOSE OF this presentation is to discuss the development, properties and characteristics of wash primers so that they may be considered as a principle for future developments. Because of the urgency of the project during World War II, little time was allowed for detailed study of the fundamental wash-primer reaction. Wash primers were developed because of a need for a material possessing certain fundamental properties, i.e. improved adhesion of protective coatings to metal and better resistance to corrosion.

Subsequently, several independent investigators^{1,2,3} have published data on wash-primer reactions and the products formed at the metal-wash primer interface. Data tend to confirm the existence of some of the properties predicted at the time of their development.

Wash primers were announced to the public in 1947. Thus, they comprise a relatively new method for combating corrosion of metals and improving adhesion of protective coatings. Wash primers resulted from work done by Bakelite Company laboratories during World War II under an agreement with the National Defense Research Committee. The basic patent on wash primers (U. S. 2,525,107) issued to L. R. Whiting and P. F. Wanger in 1950 was assigned to the Secretary of the Navy.

Although this project was not made available for commercial use until 1946, wash primers gained approval rapidly and are now used on an international scale. The importance of their development is widely recognized.

Terms Defined

The term "wash primers" was selected to designate a specific product that combines the properties of the familiar inhibitive wash coat or metal conditioner with the properties of the conventional anti-corrosive primer. The essential components of wash primers are phosphoric acid, chromate pigment and a polyvinyl acetal resin.

Several varieties of wash primers were developed, the first of which is called the reactive type; others are called non-reactive and reacted. The one most commonly used is the reactive type, designated WP-1 or MIL-P-15328.

The reacted and non-reactive primers to be discussed later are one-package systems but do not adhere as well as the two-package reactive wash primer. Wash



About
the
Author

L. ROBERT WHITING—A member of several NACE technical committees concerned with surface preparation and organic protective coatings, he formerly was chairman of T-6G. With Bakelite Company, Division, Union Carbide Corp. he has worked on polymer chemistry and coatings development. During World War II he worked on government projects in developing anti-corrosive and anti-fouling coatings. He is now with Bakelite's New Products Engineering Dept. A graduate of University of Kansas, 1921, he worked from 1921 to 1924 in the petroleum industry as a chemist, before joining Bakelite.

primers can be formulated that are equally effective over iron, steel, aluminum, copper, zinc and a wide variety of other metals.

The intricacies of the complex chemical reactions are not all known, but generally the function is manifested in three ways: by forming an inorganic and organic film on a metal surface, by preventing or retarding corrosion and by providing a base that improves adhesion and integrity of protective coating systems.

The inorganic-organic complex formed by the wash-primer reaction results from a series of complicated reactions that must occur in correct relationship to one another. The inorganic and organic films cannot be separated by any feasible method. The reactive wash primer and the metal to which it is applied become an integral part of the system at the interface. All attempts to remove the coating by conventional methods have resulted in partial removal of the metal substrate.

This paper is devoted principally to the reactive wash primer since it is the most widely known, provides the highest degree of adhesion and is most effective in retarding corrosion. The formula is shown in Table 1.

The acid diluent is added to the base grind just prior to use. This mixture is stable for about 24 hours and can be applied by brush, spray, dip or other conventional methods.

Abstract

Development, characteristics and properties of wash primers are explained. Data are given on wash primers principally because of their efficiency and wide use. Reactive and non-reactive types resulted from efforts to understand better the reactions involved and as a result of efforts to produce a one-package system.

Reacted wash primers are made by reacting phosphoric acid and chromium oxide in a solution of butyral resin at high temperature, producing a clear green lacquer. Non-reactive primers are prepared by dispersing chromates of lead, strontium and barium in a solution of butyral resin and then adding phosphoric acid. A stable wash primer was prepared by dispersing chromic phosphate in a solution of PVB. 5.4.5

Some of the advantages of WP-1 reactive type are listed below:

1. Easily applied and dries rapidly.
2. Usable over wide range of temperature and humidity.
3. Can be applied to a variety of metals with good results.
4. Provides temporary protection until protective coating is applied.
5. Prevents or retards under-film corrosion.
6. Exhibits high degree of adhesion to metals.
7. Up-grades performance of subsequent protective coating systems.
8. Usable in coating systems that require baking at elevated temperatures.

Problem and Its Solution

For many years, metal conditioners or pretreatments have been used to improve adhesion of protective coatings to metal and to retard underfilm corrosion. Many conditioners consist of phosphoric acid, metal phosphates or a source of chromium ions and/or other ingredients. The well known industrial phosphating methods for treating metals result in an adherent, uniform coating of crystalline phosphates on the metal surface. They inhibit the spread of rust if the paint film is ruptured and minimize blistering known as alkaline peeling. Inhibitive wash coats used as dilute aqueous solutions of phosphoric acid resulted in a non-uniform, powdery deposit with little resemblance to the phosphate coating produced by the industrial process⁴ which uses a dip tank. Because aqueous phosphoric solutions can not be used at sub-freezing temperatures encountered at shipyards and because industrial processes require immersion in hot treating and rinsing solutions, a practical method was needed for treating ship hulls prior to painting.

Because chromic acid makes metals passive and because chromates used in anti-corrosive primers assist in preventing corrosion, a metal pretreatment combin-

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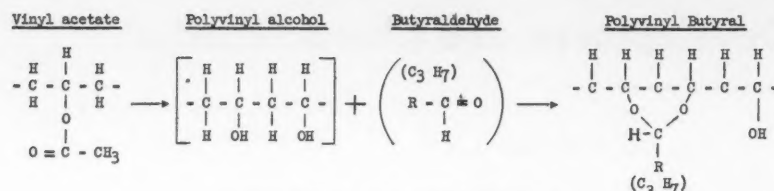


Figure 1—Schematic of the production of acetals.

ing the properties of phosphating with the passivating action of chromates would be more effective than either used alone. This was the basic idea behind the formulation of the wash primer.

A first step was an attempt to produce an improved pretreatment by the use of an alcohol solution of vinyl butyral resin containing phosphoric acid. This material could be used effectively over a wide temperature range—a necessity in outdoor painting operations. It always produced the same desirable results on the metal and provided an acceptable base for subsequent application of anti-corrosive primers. A highly effective anti-corrosive primer consisting of a solution of polyvinyl butyral pigmented with zinc tetroxochromate was formulated to obtain excellent adhesion when this butyral primer was applied over the butyral wash coat containing phosphoric acid. Certain paints based on butyral resin and copper pigments, which contained a metal ion, gelled when stored in an iron container.

The resin-metal complex formed by this gelation adhered tenaciously to the iron container and was insoluble in most solvents although polyvinyl butyral is readily soluble in alcohols. It was then postulated that a controlled gelation of polyvinyl butyral could result in a film with outstanding adhesion to metals. Thus, it was a combination of the principles involved in phosphating treatments and the chromate-pigmented butyral primer that produced the reactive wash primer.

Wash-Primer Reaction

Chromium ions not only are effective in retarding corrosion of metals but also insolubilize or cross link polyvinyl butyral. Phosphoric acid, under certain conditions, reacts with butyral and also with metal to retard corrosion. Application of the reactive wash primer to ferrous metal may result in the formation of iron phosphate, zinc phosphate, chromium phosphate and a polyvinyl butyral-chromium complex at the wash primer-metal interface.²

Zinc yellow, $\text{ZnO} \cdot \text{K}_2\text{O} \cdot 4\text{CrO}_3 \cdot 3\text{H}_2\text{O}$, described in Table 2, contains about 25 percent water-leachable ingredients.² Wash primers can be produced by using a combination of this pigment and butyrals; however, excess phosphoric is required to react with the potassium salt, and the resulting potassium phosphate detracts from the efficiency of the wash primer. Zinc tetroxochromate, ZTO ($\text{ZnCrO}_4 \cdot 4\text{Zn}(\text{OH})_2$) or $5\text{ZnO} \cdot \text{Cr}_2\text{O}_3 \cdot 4\text{H}_2\text{O}$, also described in Table 2, is produced commercially with less than one percent of water-soluble ingredients. It also furnishes zinc ions that may react with phosphoric acid to produce desirable zinc phosphate. ZTO and phosphoric acid react, producing zinc phosphate and liberating a chromate. The chromium ions also function in the insolubilization or cross linking of the butyral. Any excess chromate ions passivate the metal. The reactive wash primer always exhibits the characteristic color change from yellow to green indicating the reduction of chromium.

Phosphoric acid in contact with ferrous metals may form iron phosphates and in contact with zinc compounds may form zinc phosphate. The acid assists in the formation of the chrome-phosphate-butyral complex and with the water used in wash primers controls the pH of the mixture. Phosphoric acid does not react with polyvinyl butyral at the concentration of solvent used in the wash primer. The presence of ZTO is necessary for formation of the assumed chrome-phosphate-butyral-complex. Acids other than phosphoric produce a reaction with ZTO and butyral but do not impart the desired properties as does phosphoric. The ratio of phosphoric acid to ZTO or PVB is not critical. However, best results are obtained when the ratio of ZTO-butyral-phosphoric acid is 2:2:1. It is extremely difficult to detect the effect of a change in the amount of phosphoric acid, but a change of 25 percent is readily apparent.

Acetals are produced by hydrolyzing polyvinyl acetate to the alcohol, then reacting with an aldehyde. (See Figure 1.)

The resulting acetal is influenced by the molecular weight of the starting polyvinyl acetate, the choice of aldehyde and degree of acetalization. Sometimes referred to as PVB, the polyvinyl butyral used in wash primers contains about 20 percent polyvinyl alcohol in the molecule. The reactive group necessary for cross-linking is the hydroxyl group. Vinyl polymers such as the homopolymer of vinyl chloride contain no reactive group, a factor that may account for the difficulty in producing reactive wash primers with this polymer.

When laboratory grade 85 percent phosphoric acid is added to the base grind of WP-1, gelation usually occurs immediately. When the acid was diluted with approximately equal parts of water and slowly added to the base with mild agitation, a stable mixture could be obtained. This mixture, if used within 24 hours, exhibits good adhesion to metal after the solvents evaporate and excellent adhesion after aging a few days. This adhesion develops even though the dried film is immediately coated with an anti-corrosive primer.

PVB undergoes thermal degradation at moderate temperatures of about 350 F. The PVB complex formed in the wash-primer reaction has considerably more heat resistance. WP-1 used under a finish requiring several hours baking at 500 F still retains its adhesion and integrity—good evidence of crosslinking.

After the mechanism of the wash-primer reaction was studied, the wash-primer system was found to provide more than one approach to preventing corrosion.² First, the system produces $\gamma\text{Fe}_2\text{O}_3$ and a zinc phosphate film similar to that produced in various phosphating treatments. Phosphate treatments of this kind are not always satisfactory because they exhibit porosity. Machu³ suggests treatment of these films with chromates. The function of the chromate, according to Mayne and Pryor,⁴ is to patch up any holes in the phosphate film by oxidizing the exposed metal to $\gamma\text{Fe}_2\text{O}_3$. Evans⁵ points out that it is inadvisable to use chromates as inhibitors in insufficient quantities. Bloom² further states that wash primers take care of this by providing a steady stream of chromate ions from the slowly dissolving unreacted ZTO. The polymeric film, of course, also acts as good mechanical protection for the metal surface and serves as a base for good paint anchorage.

One of the functions of an anti-corrosive coating is to prevent water and cor-

TABLE 1—Formulation of WP-1 Reactive Type Wash Primer

Base Grind	Parts by Weight	Acid Diluent	Parts by Weight
Polyvinyl butyral . . .	7.2	Phosphoric acid.	3.6
Basic zinc chromate . . .	6.9	Water	3.2
Extender	1.0	Isopropanol	13.2
Carbon black.	.1		20.0
Isopropanol . . .	48.7		
Butanol	16.1		
	80.0		

TABLE 2—Reactive Agents in Wash Primer

Zinc Yellow— $4\text{ZnO} \cdot \text{K}_2\text{O} \cdot 4\text{CrO}_3 \cdot 3\text{H}_2\text{O}$			Zinc Tetroxochromate $5\text{ZnO} \cdot \text{Cr}_2\text{O}_3 \cdot 4\text{H}_2\text{O}$ or $\text{ZnCrO}_4 \cdot \text{Zn}(\text{OH})_2$	
	Percent ¹	Percent ²	Percent ³	Percent ⁴
ZnO	37.2	36—40	36.8—39.2	70.3
CrO ₃	45.8	36—48	41.3—43.6	17.3
K ₂ O	10.8	—	10.4—11.5	—
H ₂ O	6.2	—	6.1—7.3	12.4
Water soluble salts	—	18—34	(1.3—3.3 SO ₄ as SO ₃)	100.0
	100.0			

1. Theoretical

2. R.W. Leisy, U. S. Pat. 2,251,846, 1941

3. Brizzolara et al. *Ind Eng Chem*, 29, 656 (1937)

4. Cole and LeBrocq, *J App Chem*, 5, 149 (1955)

rosive ions from coming in contact with the metal surface. By use of electrolytic resistance measurements, Bacon, Smith and Rugg⁹ showed that a difference in protection can be expected from a vinyl red-lead anti-corrosive primer with and without the wash primer. The anti-corrosive primer alone at 3.2 mils thickness showed a steady decrease in protection during 25 days immersion in sea water. The same primer applied to a film thickness of 2.9 mils over 0.5 mils of wash primer indicated no appreciable drop in resistance in 50 days. The total film thickness was approximately the same in both cases; therefore, it can be concluded that the wash primer retards or prevents migration of water and ions that cause corrosion reactions at the metal interface and detract from film integrity.

Most of these comments apply only to the reactive-type wash primer. Other types were prepared in the attempts to achieve a better understanding of the reaction and to produce an effective one-package system.

TABLE 3—Reacted Type Wash Primer
XL 5367

Solution		Parts by Weight
A	Polyvinyl butyral.....	11.0
	Denatured alcohol.....	62.1
		73.1
B	Chromium trioxide.....	.46
	Water.....	94
		1.4
C	Phosphoric acid (85%).....	1.1
	Acetone.....	9.9
		11.0
Thinner	n-Butanol.....	14.5
		14.5
		100.0

Heat Solution A to 50°C. Solution B is slowly added to Solution C; this mixture is then added to Solution A and held at 50°C. for 25 min., cool (and add) n-Butanol. The resulting product is stable for many months.

TABLE 4—Non-Reactive Type Wash Primers

	XE 5220	XE 5298
Base Grind	Parts by Weight	Parts by Weight
Polyvinyl butyral.....	9.0	9.0
Chromic phosphate.....	9.0
Lead chromate.....	8.6
Ethanol.....	54.5
Extender.....	1.4	1.4
Isopropanol (99%).....	53.0
Methyl isobutyl ketone.....	13.0	16.1
	85.0	90.0
Acid Diluent		
Phosphoric acid (85%).....	2.9	1.8
Water.....	2.9	1.8
Isopropanol (99%).....	9.2	6.4
	15.0	10.0
	100.0	100.0

The acid diluent is added to the base grind. Both wash primers are stable for many months.

Reacted and Non-Reactive Wash Primers

First of these one-package systems formulated, a reacted wash primer, is described in Table 3. From the assumption that a chrome-phosphate-butyral complex played an important role in wash primer performance, attempts were made to produce a reacted wash primer forming this complex, in situ. Chromium trioxide, phosphoric acid and a solution of butyral were reacted at elevated temperatures. A clear green lacquer was formed that eventually found use as a coating for chromium and aluminum foils and in other commercial applications. The film did not insolubilize when applied on metal nor exhibit the same amount of adhesion as the reactive WP-1. This may be due to the reduction of chromium before it comes in contact with metal or to the absence of zinc ions prohibiting the formation of zinc phosphate.

Non-reactive wash primers (Table 4) were produced by pigmenting a solution of PVB with various chrome-bearing pigments. Chromic phosphate was assumed to form in the reactive wash primer. As a result, the use of chromic phosphate as a pigment was investigated. Chromic phosphate was ball milled into the PVB solution and phosphoric acid added. Here again no insolubilization of the film on metal occurred, neither was the excellent degree of adhesion obtained. A fair-to-good anti-corrosive primer resulted by replacing chromic phosphate with lead chromate, but this innovation found limited commercial use. However, this wash primer did give good results in fresh water. Chromates including those of barium, strontium and combinations thereof were evaluated and resulted in stable wash primers with certain desirable properties. However, none of these approached WP-1 in over-all properties.

Absence of the insolubilizing or cross-linking in films of wash primers from those other than the reactive type is demonstrated easily by coating steel panels with wash primers and immersing the coated panels in butanol. A WP-1 film that has been allowed to dry only a few hours is softened by the solvent and easily removed but an aged WP-1 film is softened very little, if at all. An unpigmented film is readily softened.

General Recommendations

Condition of the metal surface to which wash primers is to be applied influences ultimate performance. Removal of scale, dirt, grease, oil and loose rust gives better performance of any paint system. Sandblasting of metal before applying wash primer is not essential, but all contamination and loose rust must be removed. Tightly adhering mill scale or a small amount of rust is not too objectionable. But of course, there is no substitute for a clean metal surface if best results are to be obtained.

One peculiarity of the reactive wash primer not satisfactorily explained is the critical limitation of the effective film thickness. Indications are that the thinnest coating that forms a continuous film is adequate. Films of over one mil thickness do not develop toughness and adhe-

sion. For most applications, a dry-film thickness of approximately 0.5 mil is recommended.

Use of metal pretreatments usually detracts from wash primer adhesion; an exception is the sodium nitrite and diammonium phosphate treatment used in the wet sandblasting process. Use of dilute acetic acid on galvanized iron results in poor adhesion of the wash primer. All these properties are obvious when it is recalled that the wash primer reacts with the metal. If there is a barrier of any kind such as the products of pretreatments or contamination between the wash primer and the metal, little if any reaction can occur. Wash primers will adhere to mill scale. However, if the mill scale is loosely adhering to the metal, the mill scale will fall off taking the wash primer with it, and the coating system gets credit for the failure.

In conclusion, data have been presented to show that a combination of metal pretreatment and anti-corrosive coating is available and is prepared by utilizing the well known properties of phosphates and chromates in conjunction with a binder of high integrity. Future developments may produce a stable one-package system that could be applied effectively in one coat at a film thickness adequate to serve as a complete anti-corrosive system. The present wash primer has been shown to develop unusually high adhesion to a variety of metals. When followed by an efficient protective coating system, this primer exhibits remarkable protection of the metal. The film insolubilizes at ambient temperatures and improves adhesion and integrity of most coating systems. The value of protective coating systems based on wash primers has been demonstrated by years of service in the marine, chemical and other industries.

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Discussion by Harold C. Wells, Los Angeles, Cal.:

Can wash primers be applied over moist surfaces?

Reply by L. R. Whiting:

Yes. After water bleeds through, adhesion and reaction will develop to give excellent surface.

Discussion by William F. Gross, Arabian American Oil Co., New York City:

Under what generic types of primers are wash primers unsatisfactory, due to presence of residual acid?

Reply by L. R. Whiting:

Wash primers are not recommended under catalytically-cured epoxy coatings. Members of the audience remarked that poor experiences had resulted from their use under neoprene paints and Saran-based coatings.

Discussion by Stanley Roth, Dow Chemical Co., Midland, Michigan:

Answer to the question "Do Saran coatings adhere to wash primers?" In the Navy's use and in our own laboratories, it has been found that the Sarans do not adhere to wash primers. In this respect they are similar to the top-coat type vinyls.

Discussion by Vance N. Jenkins, American Petroleum Institute, New York City:

In England it apparently is customary for several epoxy systems to be applied over wash primers. Better adhesion and protection are claimed to result. I understand that the U. S. Navy does not recommend Saran coatings be applied over wash primers. Does anyone have any direct experience that would indicate why this is not favored?

Reply by L. R. Whiting:

W. W. Cranmer, Material Test Lab., Bldg. 121, U. S. Naval Shipyard, Philadelphia, Pa., has data on this point.

Discussion by Austin K. Long, Glidden Company, Cleveland, Ohio:

The use of catalyzed epoxy systems over wash primers is not recommended since there is loss of adhesion under accelerated salt spray tests. Also neoprene coatings have no adhesion over WP-1 primer although there may be heavily modified neoprenes which may be successful.

Discussion by Joe E. Rench, Napko Corp., Houston, Texas:

In answer to the question "Has anyone any experience with the use of chlorinated rubber over wash primers?" Yes, our all-chlorinated rubber systems incorporate the wash primer and have been used with excellent results over the past 8 to 10 years. The all-chlorinated systems have been used without inhibitive pigment in the coating used immediately over the wash primer particularly in areas of extreme chemical exposure. The results have been excellent.

Discussion by John J. Corely, General Electric Co., Richmond, Washington:

You mention the advisability of not delaying more than 24 hours before applying the next coat. Does the same timing apply to laying adjacent strips of the wash primer itself?

Discussion by W. A. Severance, Cleveland, Ohio:

In answer to the question "What is the effect of WP aging on adhesion of top coats?"—Too long aging in the pot after mixing results in good primer adhesion but may result in poor top coat adhesion. In reacting, the primer insolubilizes so that solvents will not bite in. After applying, top coat can be applied in as little as 10 minutes but should be earlier than 24 to 48 hours, depending on atmospheric conditions.

Reply by L. R. Whiting:

Too long aging after mixing distracts from adhesion of wash primer to metal. Age of wash primer film does not affect the adhesion of vinyls and many other top coats.

Discussion by Kenneth E. Wright, Arlington, Virginia:

Wash primers on pre-treatment coatings should not be used under coal-tar emulsions or under primer and hot enamel but could be used under cold applied coal tar. However, our experience indicates no particular advantage. What has been the experience of others in the use of wash primers under bituminous type coatings?

Reply by L. R. Whiting:

Dr. Fair of Koppers has data on this subject.

Any discussion of this article not published above will appear in the December, 1959 issue

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Evaluation of Protective Coatings for Ship Bottoms*

By J. R. BROWN

Introduction

PROTECTIVE COATING technology for ship bottoms has made great strides during the last few years. These advances, together with the use of cathodic protection have enabled the Royal Canadian Navy to extend the period between drydockings from six months, as recently as 1947,¹ to a present maximum of two years. With these advances has come an ever increasing need for a reliable and rapid method of evaluating bottom coatings.

In general, a practical evaluation system must meet the following requirements:

1. Control of surface preparation, application and exposure conditions.
2. Direct correlation of exposure conditions with service.
3. Rapidity.

If these are achieved, the result will accurately predict coating behavior under service conditions. Examination of a ship trial's and panel trial's ability to meet these requirements is of value.

Limitations of Ship Trials

A ship trial must be performed under prevailing weather conditions and with the surface preparation and application provided by the workmen. These factors are subject to meager control in the drydock and differ for each drydocking and from time to time during each drydocking. Because the effect of these variables may outweigh differences in the capabilities of coatings under comparison, ship scale trials cannot be expected to give truly comparative results. Comparison of two paints on the same ship bottom by quartering will reduce but not eliminate these errors.

The extent of this problem can be represented by a hypothetical plot as shown in Figure 1. An ideal coating would give performance independent of drydock conditions as represented by Curve A. How the performance of a non-ideal coating is affected by drydock conditions is a subject for conjecture. All coatings may vary in the same manner with variation in these conditions, in which case their curves will have the same slopes and the same origin. The extent to which one coating is better than another could be ascertained by comparing their performance when further improvement in conditions does not further improve performance. Such a case is illustrated by Curves B and D.

In the more general case, the origins would differ from coating to coating, as illustrated. However, it is likely that the slope as well as the origin will vary, in which case it will be necessary to com-



About
the
Author

J. R. BROWN has been employed since 1953 by the Defense Research Board of Canada at its Pacific Naval Laboratory in Esquimalt, B. C., where he is engaged in laboratory and field studies of ship bottom coatings. He has a BA and MS in organic chemistry from the University of British Columbia.

pare curves such as B, C and D to curves such as E. For purpose of this discussion, coatings with the characteristics of Curves B and C can be compared. Coating B would be superior to coating C. However, if the conditions on application of the first system were those noted by the dotted line and those for the second system by the dash line in Figure 1, an erroneous comparison would result. Thus, not only must comparative evaluations be conducted under standard conditions but also these conditions must span the range of conditions experienced in the drydock.

As an example of the lack of control typical of drydock painting, a recent ship trial directed by the author can be cited. The ship was divided into six sections to compare three different systems. The discrepancies noted were as follows:

1. A complete extra coat was applied to one section in error.
2. A conscientious painter was discovered touching up some areas of one section because it looked thin and he had paint left over.

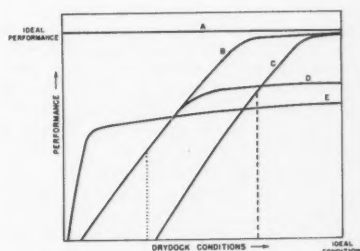


Figure 1—Probable relationships between drydock conditions and coating performance. Line A represents coating performance independent of drydock conditions. Curves B, C, D and E represent different coatings.

Abstract

Requirements for a practical system to evaluate ship bottom coatings are listed. Panel trials are shown to be better than service trials for coating evaluations.

Application of the d-c technique of coating evaluation is illustrated by comparison of three coating systems in static immersion and by a study of the influence of number of coats on performance.

Investigation of drydock variables shows the reduction in coating performance resulting from rusting and the detrimental effect of 5 percent H_2PO_4 as an inhibitor after sandblasting. 2.1.2

3. At a later date, in repainting after sandwashing,* two sections allocated to one type paint received an extra coat through misinterpretation of instructions.

These discrepancies occurred in the variable which is probably the most easily controlled, the number of coats. Since their discovery was purely fortuitous, there probably were others undetected. Results of this trial could not, then, reflect the relative capabilities of the coatings under evaluation.

Exposure conditions provided by a ship in service probably meet the test requirement, at least within each class of ship. However, when the ship is cathodically protected, the environment is partly under the control of the operator of the system. Therefore it cannot be considered standard.

The time required for a ship trial must be the interval between drydockings.

Limitations of Panel Trials

Panel exposure for evaluating bottom coatings has the advantage that it allows adequate control of surface preparation and application conditions. Since replication is simple, influence of the variety of surface preparation, application and weather conditions experienced in the drydock can be studied simply by varying

* Sandwashing is defined as the removal by sandblasting equipment of all loose paint, corrosion products and other foreign matter. The surface thus treated is ready for repainting.

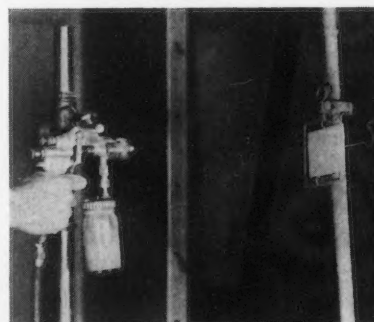


Figure 2—Pendulum spray apparatus. Panels were mounted on the pendulum and the spray gun on a pillar stand to ensure uniform and reproducible application conditions.

*Submitted for publication September 22, 1958.

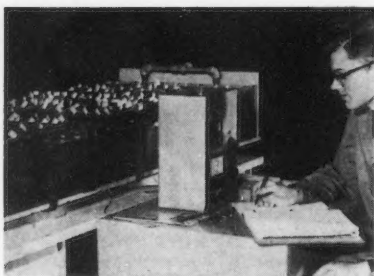


Figure 3—Static immersion tank and d-c resistance meter. Prepared panels are suspended from glass sheathed rods in the tank of fresh running sea water. The instrument shown was used to measure potential difference and d-c resistance.

these conditions in the laboratory. Similar studies using ships would be unwieldy because of the number of ships required. However, the exposure condition is not directly related to service because the velocity effect is ignored.

In addition, because the panels are not subjected to the effect of rapidly moving water, the time required for evaluation is likely to be even longer than that for a ship trial, other things being equal, although some time reduction can be achieved by reducing the number of coats applied.

An important limitation common to both ship and panel trials is that they depend on visual examination for their evaluation. Therefore, evaluation can be only qualitative at best.

In summary, service trials are not adequate for evaluating bottom coatings; they fail to meet all three major requirements. Panel trials, however, have the advantage that control and replication is possible. It is necessary, then to provide for exposure of the panels to high velocity sea water and to measure coating condition instrumentally to eliminate errors of visual measurement. In addition to improving precision of evaluation, a method using instruments would reduce the time required. These requirements fulfilled, a range of standard conditions for surface preparation must be devised to show the effect on each coating system of the major variables operative in the drydock.

This paper discusses an attempt to devise an evaluation system to meet these requirements.

Experimental Procedures

Preparation and Exposure

Mild steel panels measuring 3 inches x 4 inches x 1/8 inch and fitted with question-mark shaped hooks were wet sandblasted with 80-mesh alumina, rinsed in hot water and allowed to dry. Coatings were applied by spray to the maximum thickness possible short of sagging. To ensure uniform and reproducible application conditions, panels were mounted on a pendulum and the spray gun on a pillar stand (Figure 2). To eliminate edge effects, panel edges and stems were painted by brush with several more coats than the faces.

The prepared panels were suspended from glass sheathed rods in a tank of fresh running sea water as seen in Fig-

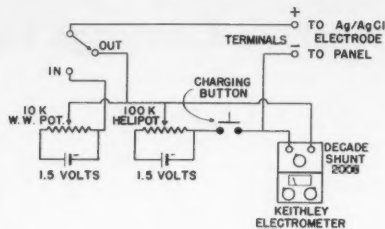


Figure 4—Circuit schematic of the d-c resistance meter used.

ure 3. The suspension hooks were kept clean and greased during exposure and were separated from the glass rods by a clean lucite spacer while electrical measurements were being taken. These precautions were necessary to prevent salt bridging between panels or down the length of the suspension hook.

Electrical Measurements

Capacitance and a-c resistance were measured at 1000 cycles using a Brown Impedance Bridge Model 250-C1. The cell consisted of the panel under examination and a silver-silver chloride electrode. For potential difference and d-c resistance measurements, a Kiethley Electrometer Model 200 fitted with a Kiethley Shunt Box Model 2008 was used. This instrument is simpler to operate than that used by Bacon et al.² and sufficiently accurate for the purpose. Two potentiometers, each supplied by a 1 1/2-volt dry cell, were included in the circuit, the first to provide added potential for resistance measurement over 10⁸ ohms and the second to charge panels having a long time constant, that is, those with a resistance of about 10¹⁰ ohms. The cell consisted of the test panel as anode and a silver-silver chloride electrode as cathode. The instrument is shown in Figure 3, and a diagram of the circuit is given in Figure 4. Measured open circuit and shunted potential differences provide the resistance according to the formula:

$$R = R_s \left(\frac{E_o}{E_s} - 1 \right) \quad (1)$$

where R_s = shunt resistance

E_o = open circuit potential

E_s = shunted circuit potential

Special Requirements

Exposure Conditions

The effect of velocity on coating performance has been investigated in several laboratories^{3,4} where it has been found that the rate of breakdown is accelerated over that for static immersion. This has been confirmed at the author's laboratory, at least for a bituminous system. The machine used consisted of a 36-inch diameter disc with provision for panels to be mounted in the plane of the disc at the periphery. This assembly was rotated in a sea water tank with a peripheral velocity of 15 knots. However, the requirement for cathodic protection during exposure and for taking electrical meas-

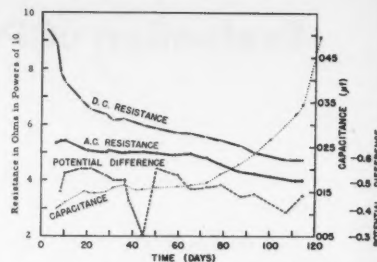


Figure 5—Comparison of the electrolytic properties of a coating.

urements has necessitated a change to a water tunnel type apparatus in which the panels are stationary and the water propelled. This machine is now being built and will be operating in the near future.

It is also necessary, particularly in considering bottom coatings for Royal Canadian Naval Ships, all of which are cathodically protected, to include cathodic protection in the exposure environment. The RCN practice is to maintain the hull potential at 0.82 to 0.84 volts versus a remote silver-silver chloride electrode. The potential probably may rise to about one volt in the vicinity of the anodes. This potential has been selected as the standard for evaluating the influence of cathodic protection and has been incorporated into the exposure environment. The current required to maintain a constant potential may be taken as an indication of coating condition although it is not as sensitive a technique as the one to be discussed in following sections.

Determination of Coating Condition

References to the correlation of capacitance and a-c resistance (conductance)⁵ d-c resistance² and potential⁶ of paint films with protectivity are abundant in the literature. These properties were investigated to determine their utility as detectors in a routine evaluation system.

In theory the capacitance of a coated panel is initially a function of the thickness and type of paint modified with time by water absorption. However, when a film break occurs, it will be accompanied by a sharp capacitance increase which is then a function of the thin ionic double layer. The point in the plot of capacitance versus time, at which the sharp rise occurs, indicates the onset of significant film degradation. Thus, from a protective point of view, a single point of significance is obtained through measurement of capacitance. The time for degradation to proceed to this point must be awaited before any information is gained. Also, the break, when it occurs in practice, is often a gradual rather than a sharp transition, and the result, therefore, is indeterminate.

A typical capacitance-time curve is given in Figure 5, which also shows the potential and a-c and d-c resistance behavior of the same panel.

As for the relationship between panel potential and coating condition, bare

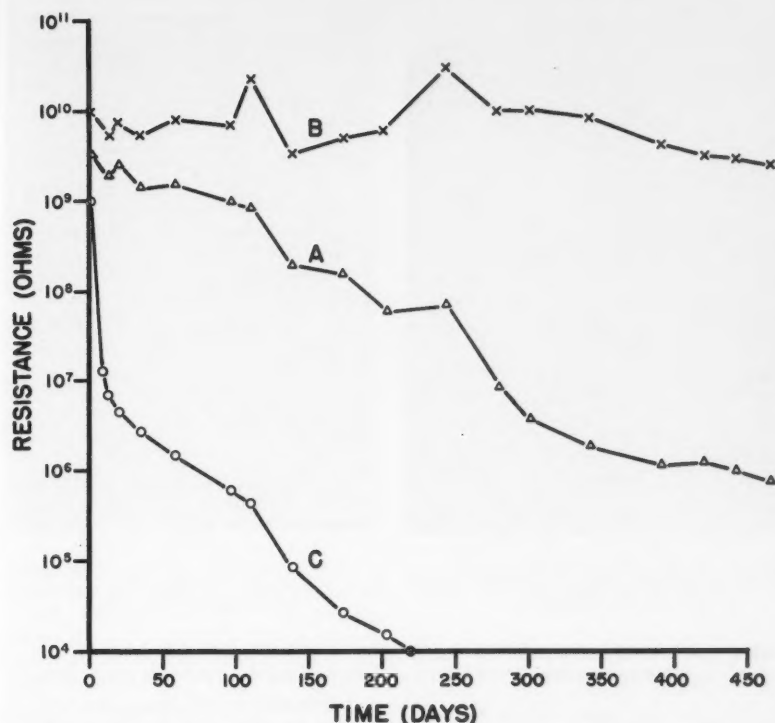


Figure 6—Comparison of three coating systems in static immersion using the d-c resistance method. Curve A is a coating system using one coat each of wash primer, anti-corrosive primer, intermediate primer and anti-fouling. Curve B is the same system as Curve A except that intermediate primer coat is replaced by a second anti-corrosive primer coat. Curve C is a coating system using one coat of wash primer, two of anti-corrosive primer and one of anti-fouling—all of a different proprietary system from Curves A and B.

steel has a potential more anodic than painted steel.^{1,7} It is therefore, not unreasonable to expect some sort of relationship to exist. Such a relationship has been found by other workers, but under the measurement conditions used in this laboratory, no correlation has been observed.

It has been shown that the fundamental property of a protective coating which limits the corrosion of the substrate is its permeability to ions, that is, its electrolytic resistance. The resistance of a coated panel immersed in sea water should, therefore, be a direct measure of the protective condition of the coating. This relationship has been verified by Bacon, Smith and Rugg² using a d-c technique and by Wormwell and Brasher³ using 1000 cycle a-c.

A comparison of the two resistance measuring techniques (Figure 5) indicates that the initial plateau at about 10⁹ ohms in the plot of a-c resistance versus time represents a limiting measurable resistance. The limit is a function of the power factor of the coating material and the frequency of the measuring apparatus. The break in the curve represents the point at which coating resistance falls below the limiting value. By chance, this value corresponds to that at which the capacitance begins to increase. In contrast, the limit on measurable d-c resistance, imposed by the time constant of the circuit, is about 10¹¹ ohms. With this higher limit a great deal of information is gained by this technique before any information is available from either the a-c resistance or the capacitance data.

When the coating resistance falls below

the critical value for a-c measurement (at about 70 days in Figure 5), the a-c and d-c plots become closely parallel although they do not coincide. Below the limiting value, the a-c plot can be assumed to represent the true electrolytic resistance. The displacement of the d-c resistance plot to higher values probably results from polarization which occurs during the d-c measurements. Because the displacement is small and almost constant, such polarization as occurs during d-c measurements does not significantly influence the results.

Application of the d-c Technique Evaluation of Coatings in Static Immersion

Figure 6 illustrates the use of the d-c resistance technique in a comparison of three coating systems on sandblasted steel. These are bituminous type coatings with aluminum flake pigment in the anti-corrosive primer. Curve A represents a system consisting of one coat of wash primer, one of anti-corrosive primer, one of intermediate primer and one coat of anti-fouling. Curve B represents the same system except that the intermediate primer has been replaced by a second coat of anti-corrosive primer. Curve C utilized a different proprietary system consisting of one coat of wash primer, two of anti-corrosive primer and one coat of anti-fouling. Each system was exposed in triplicate and the results averaged.

Superiority of the first two systems over System C was apparent within a few days; differentiation between B and A was



Figure 7—Test panel condition before painting. Panel was given Treatment No. 1: 6 dips in sea water.

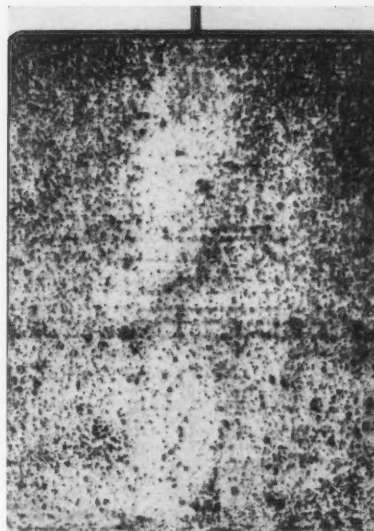


Figure 8—Test panel condition after Treatment No. 2: 1 dip in fresh water, 2-day outdoor exposure before painting.

achieved within 100 to 150 days. (Prior to this time, the difference between the two was smaller than the probable error of about one order.)

Visually, signs of coating breakdown do not appear until panel resistance has fallen below 10⁹ ohms (10⁹ ohms per sq cm). As a result, the first visual distinction between coating C and the others appeared at about 150 days. Visual distinction between A and B is still awaited (465 days).

Comparison between A and C has been confirmed in a ship trial which took 14 months. After that period the difference between the two systems was small enough to make visual comparison uncertain. By comparison, the data obtained in the laboratory permitted clear differentiation. In a service trial, difference between A and B probably would be obscured by the other variables.

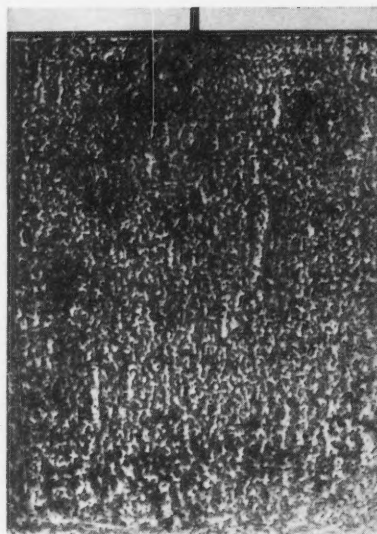


Figure 9—Test panel condition after Treatment No. 3: 1 dip in sea water, 2-day outdoor exposure before painting.



Figure 11—Test panel condition after Treatment No. 8: 1 dip in 5 percent H_2PO_4 , 1-day outdoor exposure before painting.

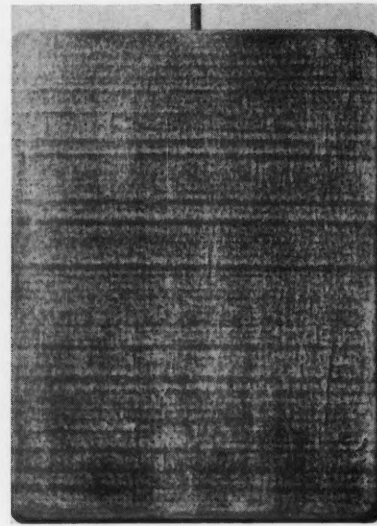


Figure 12—Test panel condition after Treatment No. 9: 3 dips in sea water. Horizontal lines resulted from rolling mill imperfections.



Figure 10—Test panel condition after Treatment No. 7: 1 dip in fresh water, 1-day outdoor exposure before painting.

These three systems also have been compared by the d-c method while under cathodic protection at a potential of one volt versus Ag/AgCl. Their performances were unaffected by this additional influence.

In Table 1 are shown performance figures for several variations of System C in static immersion. The table was produced by reading the log resistance for each panel at 30 and 60 days (R_{30} and R_{60}) and recording the average for each set. The sum of R_{30} and R_{60} is presented in the last column as a number (in arbitrary units) representative of the relative performance. A marked improvement accompanies added coats. It is also noteworthy that a coat of wash primer results in greater improvement than an added coat of anti-corrosive.

TABLE 1—Performance Figures for Variations of System C

System	Log R 30 Days	Log R 60 Days	R 30 + R 60
2 A/C, 1 A/F.....	5.0	4.0	9.0
WP, 2 A/C, 1 A/F.....	6.1	5.7	11.8
3 A/C, 1 A/F.....	5.8	5.0	10.8
2 A/C, 2 A/F.....	5.7	5.0	10.7
WP, 3 A/C, 1 A/F.....	7.2	7.1	14.3
4 A/C, 1 A/F.....	6.5	4.6	11.1
3 A/C, 2 A/F.....	6.5	5.7	12.2

A/C = Anti-corrosive.
A/F = Anti-fouling.
WP = Wash Primer.
Log R = Log Resistance.

Influence of Drydock Variables

A major drawback to ship trials is lack of control of variables operative in the drydock. To fully evaluate a coating system, the effect of these variables on system performance must be determined.

As this study's initial phase, the surface preparation was assumed to be good and that the environmental factors had their effect subsequent to this operation. This corresponds to drydock experience when the surface preparation consists of sandblasting. To evaluate the influence of surface rusting and humidity, it is then necessary only to simulate the corrosion which may take place between the time of sandblasting and painting and to maintain the test panels under the desired humidity and temperature conditions during the subsequent exposure and painting operation.

Special Panel Preparation Procedures

Standard states of rustiness were produced by three methods:

1. Repeatedly dipping the test panels in sea water, allowing the panels to dry between dips (panels dipped both three and six times were used).

2. Dipping panels once in sea water and exposing for two days to rainy weather.

3. Dipping in fresh water and also exposing for two days to rainy weather (this procedure corresponds closely to drydock procedure when wet sandblasting is used).

Because facilities for control of humidity and temperature conditions were lacking, two natural conditions were used to arrive at some indication of the importance of these variables: normal laboratory conditions which are close to ideal, and outdoor conditions during a period of rainy weather in the winter. While the latter condition would not be reproducible, it would be standard for the series. In fact, the week chosen for the outdoor exposure and painting was cool and rainy with the temperature close to 50F, and the relative humidity ranged from 70 to 100 percent—conditions generally considered poor for painting.

As a pilot run for contemplated evaluation of surface inhibiting methods, some specimens were included to evaluate the efficiency of the 5 percent phosphoric acid wash as an inhibitor of corrosion for the period between sandblasting and painting.

Coating system C as given in Figure 6 was used. This was varied in two cases by omission of the wash primer, thus providing a comparison of two different primers, wash primer and the anti-corrosive primer of System C.

In all, the series used 10 different treatments (listed below) applied in most cases to five panels each. Wet sandblasting was the initial step in all cases. The first five sets were painted indoors following the treatment described; the others were painted outdoors.

1. Six dips in sea water, allowing the panels to dry between dips.

2. One dip in fresh water followed by outdoor exposure for 2 days in rainy weather. (3 panels only.)

TABLE 2—Effect on Coating Performance of Surface Rust 5% H_2PO_4 and High Humidity

Treatment No.	R30	R60	R30 + R60
1.....	5.2	3.1	8.3
2.....	5.9	4.1	10.0
3.....	4.2	3.5	7.7
4.....	5.0	4.0	9.0
5.....	3.9	2.0	5.9
6.....	6.3	5.8	12.1
7.....	6.1	5.6	11.7
8.....	4.7	4.2	8.9
9.....	4.1	3.6	7.7
10.....	5.9-6.4	5.6-6.1	11.5-12.5

3. One dip in sea water followed by outdoor exposure for 2 days in rainy weather. (3 panels only.)

4. Freshly sandblasted, no wash primer.

5. Treatment No. 1, no wash primer.

6. Exposed outdoors one hour prior to painting outdoors.

7. Wet with fresh water, exposed outdoors 1 day prior to painting outdoors.

8. Wet with 5 percent H_2PO_4 and exposed outdoors 1 day prior to painting outdoors.

9. Same as treatment No. 1 but only three sea water dips, exposed outdoors 1 day prior to painting outdoors.

10. Same as treatment No. 1, and exposed outdoors 1 day prior to painting outdoors.

11. Normal laboratory treatment (indoor application to freshly sandblasted panels).

Figures 7 through 12 show the condition of some of the panels prior to painting.

The panels were exposed, after coating, in flowing sea water and the performance evaluated by use of the d-c resistance method.

The results of this series are listed in Table 2 and presented graphically in Figure 13. The numbers in each bar of Figure 13 refer to the treatment numbers as listed. The range of results given for Treatment No. 11 results from many repetitions in triplicate of the treatment and is given to indicate the precision of the d-c resistance method.

Discussion

Figure 13 shows that exposure of the properly cleaned substrate to high humidity before and during painting has little or no effect on subsequent performance (Treatments 6 and 7). However, application of 5 percent phosphoric acid, a rust inhibitive treatment often used to maintain the surface between preparation and painting (Treatment 8), significantly impairs performance on outdoor application. Other trials not directly comparable to the present series indicate that this inhibiting treatment does not affect coating performance when the paint is applied indoors. In consequence, reduction in performance apparently results from water retention by the hygroscopic phosphate salt layer when exposed to high humidity.

The effect of a rusty substrate on coat-

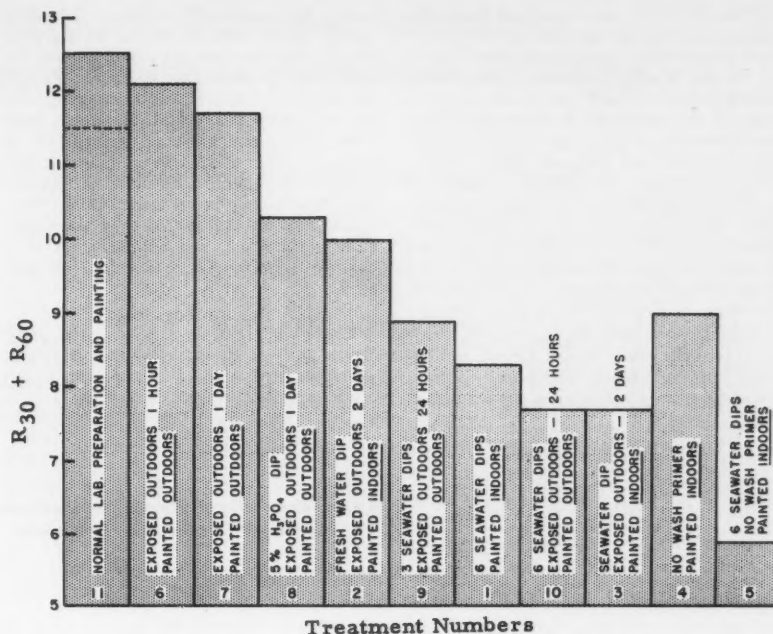


Figure 13—Bar graph showing results of the eleven treatments used on test panels. $R_{30} + R_{60}$ is the average log resistance for sets of panels at 30 and 60 days.

ing performance demonstrated by Treatments 1, 2 and 3 is severe and is roughly proportional to the degree of rusting. The 6-dip treatment (No. 1) results in too great a performance loss for use as a standard degree of rusting. The performance is reduced so low that other added variables could have little additional effect. The rusty panels exposed and painted outdoors (No. 10) gave slightly poorer performance than those painted indoors (No. 1). Though the difference between these two is not large, when it is considered that rust is slightly hygroscopic and that the performance has been reduced to such a low order by the presence of the rust that further reduction due to high humidity will be small, some significance can be attached to this reduction. That is, the performance of paint applied to rusty steel is probably further reduced when the application is made under high humidity conditions.

When Treatment No. 10 is compared to Treatment 9, which involves a rust film produced by only 3 sea water dips, it can be seen that the 3-dip system has a much milder effect than the 6-dip system. This method is probably adequate for a standard rusty condition.

A final comparison can be made involving the effect of rust on two primers: wash primer and the anticorrosive primer of Coating C. Comparison of Treatment 1 and 11, both of which used wash primer, indicates that the effect of rust reduces the performance by about $3\frac{1}{2}$ units. (To calculate this difference, the mean value of the range given for Treatment 11 was used.) Similarly, a comparison of Treatments 4 and 5, in which the primer was the anticorrosive primer of system C, indicates a performance reduction of about 3 units. That is,

there is no significant difference between the sensitivities to surface rusting of these two primers. Whether or not the added effect of high humidity would alter this comparison has not been established.

The fact that high humidity apparently affects only the first coat of paint may simplify the problem of obtaining controlled weather conditions. Exposing panels in the controlled atmosphere of a humidity cabinet may be adequate. Panels would be exposed until the equilibrium absorption of water vapor by the surface layer was reached and then would be painted immediately.

Conclusions

Of the techniques investigated for determining coating condition, the d-c resistance method is the most satisfactory. It records a property fundamental to the protective function of the coating and provides an accurate and reliable comparison between panels under evaluation. Sensitivity of the method is such that comparative evaluations can be made in about one-fifth to one-tenth the time required for visual evaluation.

In the case described, differentiation of the poor coating from the other two required less than 10 days by the d-c technique, four to five months by visual evaluation and 14 months in a service trial. Similarly, differentiation of the two good coatings required five months by the d-c resistance method, over sixteen months visually and is unlikely to be achieved by a service trial.

Furthermore, polarization which occurs during the measurement of d-c resistance is small and constant and therefore has no effect on comparative evaluations.

The effect of two drydock variables, rusting during the period between surface preparation and painting and humidity during application, have been determined for one coating system. As expected, rust on the substrate is detrimental to performance, at least in the case of System C with and without wash primer. More important, this system is unaffected by high humidity during application except when the surface is coated with phosphate salts or rust. Thus, in this case, humidity is only a secondary factor. The importance of good surface preparation, however, is emphasized by this finding.

Initial work indicates the necessity for including the effect of velocity in a complete bottom coating evaluation system.

In the over-all picture, not all require-

ments for a bottom coating evaluation system have been achieved. In particular, the requirement for rapid motion has been only superficially investigated, and the range of standard exposure conditions has not been established. However, both have been investigated sufficiently to show the necessity for their inclusion and to show that their inclusion is practical. As a result, the requirements for an evaluation system can be met by this laboratory technique, and the behavior of a coating in service can therefore be predicted.

Acknowledgments

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Evaluation of Organic Corrosion Inhibitors For Special Applications in Petroleum Refining*

By R. B. THOMPSON, R. F. STEDMAN, CHARLES WANKAT and R. C. HENRY

Introduction

THE LABORATORY evaluation of corrosion inhibitors which may be used in petroleum refining often presents difficulties. A wide variety of environments¹ function in such applications to produce corrosion of different types of metals. This paper will discuss some of the results obtained in the laboratory evaluation of corrosion inhibitors for particular locations in refineries.

In storage tanks, for example, there usually is sufficient oxygen in the aqueous bottoms for corrosion to occur under the influence of both water and oxygen. In crude distillation equipment, air is almost completely absent although a highly acid medium usually is developed by hydrolysis of the chlorides present in the crude.

An alkaline environment usually prevails in gas recovery facilities associated with catalytic cracking units, due to the evolution of ammonia and the presence of moisture. However, it also is possible to have an excess of hydrogen sulfide present, predisposing to hydrogen attack. These substances are encountered as a consequence of the cracking process and enter the gas recovery system along with all other materials present in the reactor effluent that are lighter than gasoline. In separating the more valuable hydrocarbons from the contaminants mentioned, and also from the less valuable remaining materials usually utilized as plant fuel, compression, absorption and fractionation steps are employed.

Inhibitors are on the market which, with adequate pH control, appear to give good results in oxygen-free service such as the overheads of crude distillation columns. But most of these additives do not show satisfactory rust-inhibiting properties when tested in air by the Modified ASTM Turbine Oil Corrosion Method (ASTM D-665-54, MIL-I-25017) or the Static Water Drop Test Method. This is an obvious example of laboratory tests failing to correlate with actual field experience. Undoubtedly these methods fail to show corrosion inhibition because of the widely different environments which are present during the two laboratory rusting tests and the environment present in a crude distillation column. What is needed therefore, is either a modification of the existing tests to secure the required correlation, or an entirely new test.

On occasion the refiner and the supplier of corrosion inhibitors have a mutual interest in the pure laboratory testing of corrosion inhibitors without necessarily relating the laboratory test to a specific refinery

unit. This arises when there are specifications to be met, such as the military requirements covering jet fuel and avgas and those for corrosion inhibitors themselves. The latter specification—MIL-I-25017—includes a modified ASTM Turbine Oil Corrosion Test (D-665-54) as a performance test on the inhibitor. It is difficult to get reproducibility in the laboratory with this test as it is presently written. A modification using the newly described resistance test probes² will be discussed in this paper.

Experimental

Two types of laboratory equipment were used: a reflux apparatus, wherein effort was made to simulate refinery corrosion, and a modified military test (MIL-I-25017) using electrical resistance probes. The techniques and coupon preparation covered corrosion of steel at acid pH and corrosion of Admiralty metal in alkaline environment. Both oil- and water-soluble inhibitors were studied.

Reflux Apparatus

A schematic representation of the reflux apparatus used is shown in Figure 1. The corrosion test coupon, of mild steel or Admiralty metal, is suspended from a hook by a hole drilled through one end. The liquid flowing over the test strip is the combination of water and hydrocarbon condensate flowing from the condenser, plus the inhibitor solution coming from the reservoir through a calibrated capillary.

In operation the heater is controlled by a variable autotransformer adjusted so that the reflux rate averages 25 cc per minute by preliminary measurements. Then, knowing the concentration of inhibitor solution in the reservoir, an average concentration of inhibitor flowing over the test coupon may be calculated. A provision for introducing controlled amounts of hydrogen sulfide and/or ammonia is made by capillaries sealed into a piece of 1/4 inch tubing as described by Bloch, et al.³ After a suitable running time, 10 or 20 hours, the coupon is removed, cleaned and weighed.

Procedure for Corrosion of Steel in Acid Medium

Immerse the test strip of mild steel $\frac{1}{2}$ by 6 by $\frac{1}{32}$ inch in a test tube containing approximately 6N hydrochloric acid. Heat gently in a steam bath about 10-15 minutes and then rinse with water. A satisfactory test strip should have a roughened crystalline appearance. At this point the strip should not be touched with the hands

Abstract

A laboratory test method was developed to evaluate corrosion inhibitors for use in various parts of refineries. The apparatus devised could be used for evaluation of both soluble and water-soluble corrosion inhibitors for steel at an acidic pH and for Admiralty metal inhibitors at an alkaline pH at low concentrations of oxygen. Reasonably reproducible results were obtained with water-soluble inhibitors on steel at mildly acid pH. Reproducibility was poorer on Admiralty metal at a slightly alkaline pH. Elimination of corrosion coupons in favor of a corrosion probe gave good reproducibility.

Data reported include weight loss of strips by various cleaning methods, effectiveness of 13 inhibitors on steel in acid, corrosion rate of Admiralty metal at a pH of 8.3 (in NH_3 and H_2S), and inconsistencies in MIL-I-25017 ASTM ratings of coupons. 58.1

but instead should be handled with forceps and paper towels. Following the water rinse, the test strip should be wiped with a paper towel, rinsed with acetone and allowed to dry.

Weigh the strip to the nearest tenth of a milligram. Place 300 cc of Skellysolve C and 50 cc of acidic water (pH 4.5 using HCl) in the boiling flask. Prepare the inhibitor stock solution by dissolving the required amount of inhibitor in about one liter of a mixture consisting of $\frac{1}{3}$ xylene plus $\frac{2}{3}$ Skellysolve C.

Place the boiling flask, test strip and condenser in position and turn the heater to the setting which will give a reflux rate averaging 25 cc per minute. Start the flow of hydrogen sulfide through its capillary inlet system. Charge the inhibitor solution

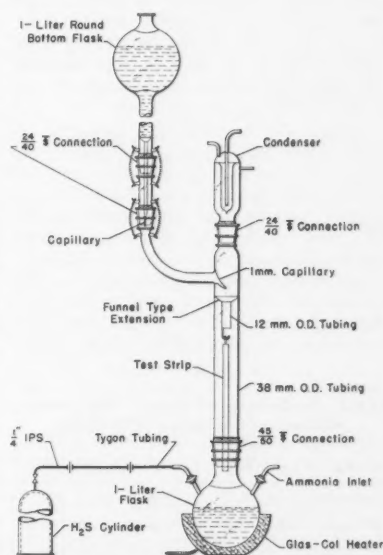


Figure 1—Schematic representation of reflux apparatus.



Thompson

Stedman

Wankat

Henry

R. B. THOMPSON received his Bachelor of Education degree from Southern Illinois University, Carbondale, Ill., in 1934. He received his PhD degree from Harvard University in 1938, and was on the staff of that university for one year working with Dr. James B. Conant. He joined Universal Oil Products Company in 1939 and is associated with their research and development laboratories. Dr. Thompson's major fields of work are with additives, isomerization and alkylation of paraffin hydrocarbons.

R. F. STEDMAN is a member of the Products Sales Department of Universal Oil Products Company, Des Plaines, Illinois. He received his BSc degree in chemistry from the University of Illinois in 1935. Mr. Stedman was engaged as chief research chemist with Cardox Corporation of Chicago and with Leonard Refineries, Inc. before coming to UOP. He is a member of NACE, and is currently employed in a technical sales capacity in the marketing of UOP's line of petroleum inhibitors.

CHARLES WANKAT has been employed at the Universal Oil Products Company research laboratories since 1936. He obtained his BSc degree in chemistry at Illinois Institute of Technology, Chicago, in 1942. Mr. Wankat has been engaged in analytical chemistry, the treating of petroleum distillates, refinery service work, and corrosion study, and is now supervisor of one of the UOP Service Laboratories at Des Plaines.

R. C. HENRY joined the research laboratories of Universal Oil Products Company in 1953. He received a BSc degree in chemistry from the University of Maryland, College Park, in 1942. After four years as Chemical Officer with the 12th and 15th AAF he did organic functional group analytical research for Shell Development Company. In 1951 Mr. Henry received a MS degree in chemistry from the University of Michigan. In 1952 he helped set up the process control laboratories at the Paducah Gaseous Diffusion Plant of the Atomic Energy Commission. Mr. Henry is currently engaged in corrosion and stability testing and analytical research for UOP.

by scouring with soap and scouring powder, then rinsing with water, and scouring again with powder alone. Rinse with water and immerse for 10-15 minutes in a 5 percent aqueous solution of sodium cyanide. Rinse the strip thoroughly in water, wipe with the paper towel, rinse again in water, followed by acetone. Allow the strip to dry and weigh to the nearest tenth milligram.

Assemble the apparatus with 50 cc of corrosive water and 300 cc of Skellysolve C in the boiling flask. Position the strip and the inhibitor stock solution in the same manner as described previously. Turn on the heat and the flow of hydrogen sulfide and ammonia and allow to operate for a period of 20 hours. Upon shutting down, immediately disassemble the apparatus and remove the Admiralty corrosion test strip. Do this quickly in order to avoid the unwanted corrosion of Admiralty metal which occurs in the presence of oxygen and ammonia.

The corroded strip is prepared for weighing by immersion in sodium cyanide solution for a 10-15 minute period, followed by a rinse with water, and immersion in dilute hydrochloric acid (about 2N) for 30 seconds. Rinse in water and repeat the cleaning cycle, starting with the sodium cyanide. It may be necessary to repeat the operations two or three times before it is possible to obtain a clean strip which is free of corrosion products. The loss of parent metal is inconsequential. Experiments with initially clean strips gave weight losses of less than 1 mg after repeating the cleaning operation several times.

Results and Discussions

Preparation of Steel Coupons

Visual classification of the corroded steel coupon was tried and found to be difficult. Ultimately a weight loss measurement was employed. In order for this to be done, the proposed cleaning procedure had to be evolved. Some of the data which led to the adoption of the final method are shown in Table 1. Note that the removal of the scale from the corroded test specimen is done with inhibited hydrochloric acid, whereas the test strip being pre-

reservoir with the one liter of inhibitor solution and put this into place. The reservoir should have an inhibitor inlet capillary which delivers one cc per minute. As mentioned previously the concentration of inhibitor flowing across the test strip can be computed from the rate of flow of the inhibitor solution plus that of the condensate.

Allow the test to run for 10 or 20 hours as the case may be. For the longer runs it may be necessary to use an inhibitor inlet capillary of decreased flow rate. In such cases it is necessary to use a higher concentration of inhibitor in the stock solution. Caution: avoid transferring even small amounts of dirt to the inhibitor reservoir flask; an extremely small amount of dirt will plug the capillary.

At the end of the desired period of operation, shut off the heat and dismantle the apparatus. Remove the test strip and immerse it briefly in a test tube full of inhibited hydrochloric acid, approximately 5N. This rapidly removes any scale which has been formed during the test. Rinse in water, then acetone, dry and weigh as described for the initial strip preparation. Since the strips were all the same size originally, and exposed for equal lengths

of time in the same manner, corrosion losses can be compared directly.

A few minor modifications are necessary in the operation when water soluble inhibitors are being studied. Start with 50 cc of the corrosive water and 300 cc of hydrocarbon in the boiling flask. The inhibitor solution charged to its reservoir is made up in water. It is advantageous to operate at an inlet rate of 0.5 cc per minute for the aqueous inhibitor solution. Cleaning and weighing of the test strip is done in the same manner as described previously.

Procedure for Corrosion of Admiralty Metal in Alkaline Medium

Clean new test strips of arsenic-inhibited Admiralty metal, 6¼ by 7⅞ by 1/16 inch,

TABLE 2—Comparison of Oil Soluble Inhibitors for Corrosion of Steel in Acidic Environment

Name of Inhibitor	Wt Percent in Stock Solution	Conc Flowing Over Test Strip, ppm	Wt Loss, mg
None.....	0	0	56, 71, 77, 74, 61, 58, 53: Ave. 64
A.....	.025	10	11
A.....	.050	20	6
B.....	.02	8	15
C.....	.02	8	13
DX.....	.02	8	11
	.05	20	2
EX.....	.025	10	8
F.....	.05	20	48
	.10	40	17
G.....	.05	20	10, 10, 11
H.....	.05	20	27
J.....	.02	8	55
K.....	.025	10	7
L.....	.025	10	44
MX.....	.025	10	49
NX.....	.05	20	19, 6

Note: Inhibitor solution added at 1 cc/min. Test duration 10 hours.

TABLE 1—Testing of Strips with Various Methods of Cleaning (10 Hr Runs)

Method of Cleaning	Wt Loss, mg
Mechanical Scouring.....	35, 28, 42, 24, 18
Mechanical Scouring and Acid..	13, 71, 33, 34, 81
Warm Acid (6 N HCl).....	56, 71, 77, 74, 61, 58, 53

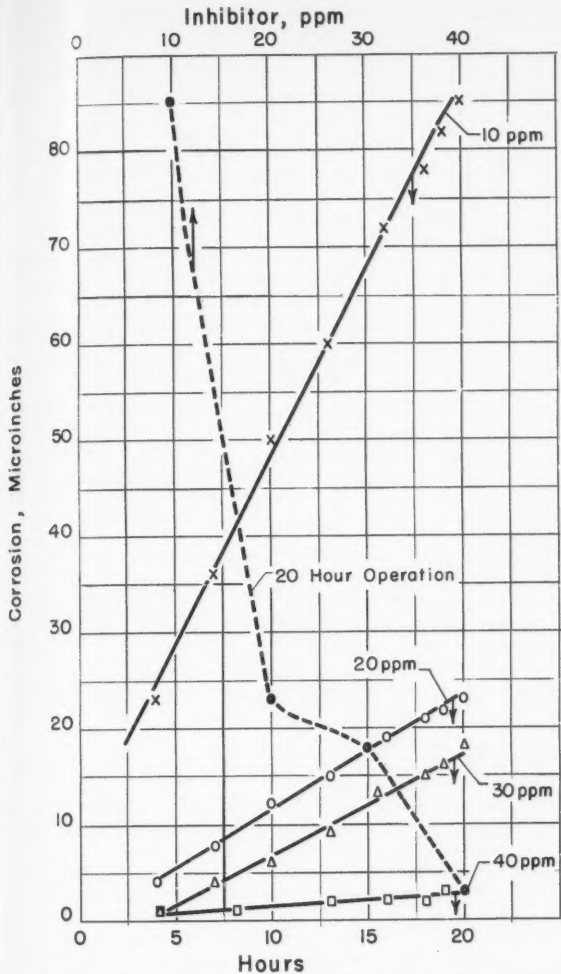


Figure 2—Corrosion rates with inhibitor DD (ASTM D-665-54; MIL-I-25017).

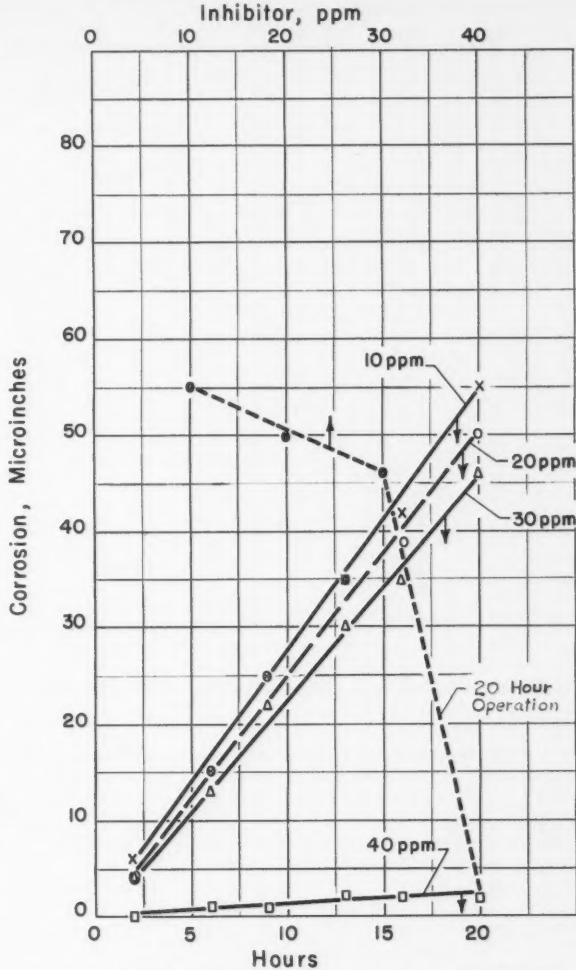


Figure 3—Corrosion rates with inhibitor G (ASTM D-665-54; MIL-I-25017).

pared for use is contacted with uninhibited hydrochloric acid.

Inhibitor Evaluation

Table 2 shows results obtained with 13 different corrosion inhibitors in a study of corrosion of steel in acidic environment. A variety of these materials appear to function as corrosion inhibitors. It is interesting that certain of the materials which are effective in the reflux test are ineffective when tested by the Turbine Oil Corrosion Method. These substances are inhibitors A, B, C, DX, EX and F. It is believed that the reflux test is much more representative of what occurs in a distillation column than is the Turbine Oil Rusting Test.

There are available at present a variety of water soluble and water dispersive corrosion inhibitors which appear to have advantages in specific applications. The data in Table 3 indicate that a good degree of protection can be obtained with several materials and that this protection can be carried down to quite small concentrations of inhibitor. Again it is found that none of the materials discussed here show any effectiveness in the ASTM Turbine Rusting Test.

TABLE 3—Comparison of Water Soluble Inhibitors for Corrosion of Steel in Acidic Environment

Name of Inhibitor	Wt Percent in Stock Solution	Conc Flowing Over Test Strip, ppm	Wt Loss, mg
None.....	0	0	36, 40, 30, 33, 28, 36; Ave. 34
F.....	0.0125 .025 .05 .10	2.5 5 10 20	9 8 6 7, 11
H.....	0.0125 .025 .05 .10	2.5 5 10 20	6 8 10 6
OX.....	0.10	20	11
PX.....	0.0125 .025 .05 .10	2.5 5 10 20	8 6 6 5, 8

Note: Inhibitor solution added at 1 cc/min. Duration of test was 10 hours.

It was of interest to study corrosion of Admiralty metal in the presence of ammonia and hydrogen sulfide because Admiralty metal condenser tubes are commonly used in gas recovery units, particularly in compressor aftercoolers. The environment here is of such ammonia

alkalinity that much corrosion frequently results.

The reflux test proved to be a useful tool for laboratory study of inhibitors. The discussion which follows is complicated by the fact that the equipment has not been fully standardized, and data are presented

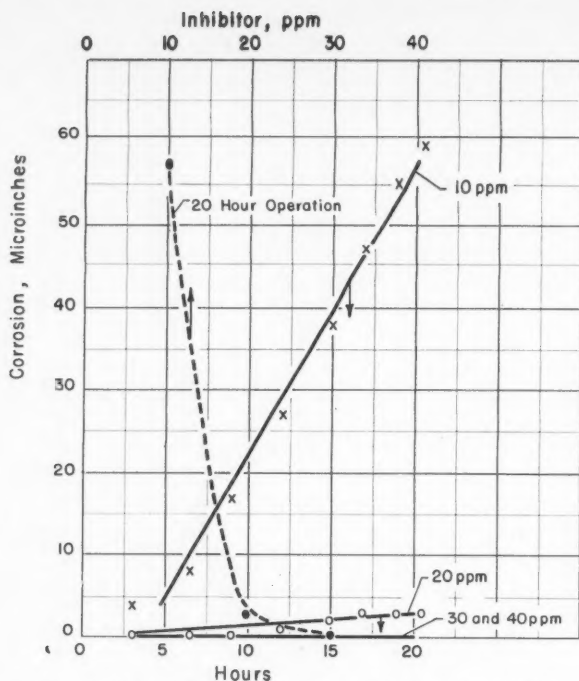


Figure 4—Corrosion rates with inhibitor I (ASTM D-665-54; MIL-I-25017).

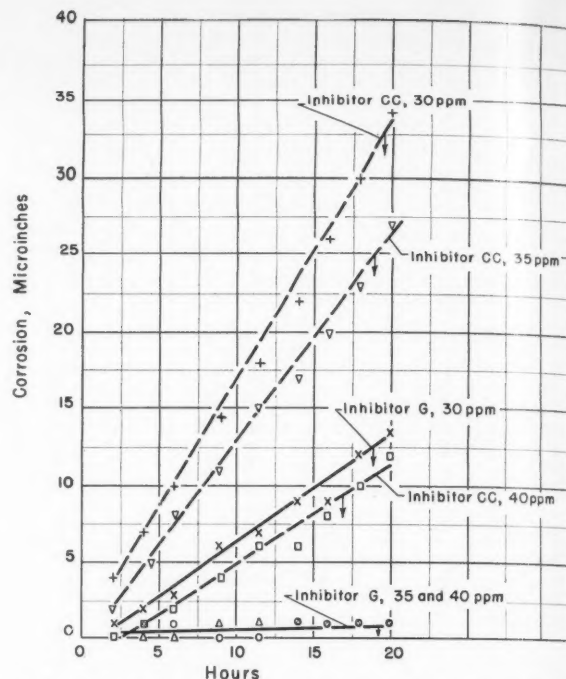


Figure 5—Corrosion rates with inhibitors CC and G (ASTM D-665-54; MIL-I-25017)

TABLE 4—Corrosion of Admiralty Metal in Medium of pH 8.3, Preliminary Runs*

Name of Inhibitor	Wt Percent in Stock Solution**	Conc Flowing Over Test Strip, ppm	Wt Loss, mg
None.....	0	0	43, 42, 43, 44, 35; Ave. 41
A.....	0.10	20	38
G.....	0.05	10	25
	.10	20	16
H.....	0.10	20	54
R.....	0.10	20	34

* This run made with set of capillaries for NH₃ and H₂S which gave low rates. Duration of run 20 hours.
 ** Inhibitor solution added at 0.5 cc/min.

TABLE 5—Corrosion of Admiralty Metal in Medium of pH 8.3, Run 2*

Name of Inhibitor	Wt Percent in Stock Solution**	Conc Flowing Over Test Strip, ppm	Wt Loss, mg
None.....	0	0	95, 105, 108, 107, 121; Ave. 107
F.....	0.10	20	66
I.....	0.05	10	23
L.....	.10	20	27
S.....	.10	20	41
T.....	.10	20	83
U.....	.10	20	37
V.....	.10	20	86
W.....	.10	20	84
Y.....	.10	20	44
Z.....	.10	20	70
AA.....	.10	20	84
BB.....	.10	20	221

* Run 2 at H₂S rate 23 cc/min. and NH₃ rate 8.3 cc/min. Duration of run 20 hours.
 ** Inhibitor solution added at 0.5 cc/min.

for three groups of runs which give somewhat different corrosion rates in the absence of inhibitors. Despite this weakness, the individual groups of runs at the separate conditions can be made reproducible and hence the results are of value.

The data in Table 4 were obtained using two particular capillaries for ammonia and hydrogen sulfide. Three of the inhibitors tested, A, G and H, are commercial additives which have a history of successful operation in the prevention of corrosion

of steel in an acid medium of 5-7 pH. This test method indicated that only one of them (inhibitor G) afforded some degree of protection, while the others were essentially ineffective.

The next series of runs were made with an ammonia capillary which allowed a much higher ammonia rate and caused much greater corrosion in the absence of inhibitors, 107 milligrams weight loss as compared to 41 milligrams weight loss for the data of Table 4. Most of the inhibitors tested proved to be ineffective; inhibitor I was the only one found to be really effective. It is interesting to note the action of inhibitor BB, a material which is a good oil-soluble detergent. These detergency properties of inhibitor BB are believed to be responsible for the great increase in corrosion which occurred with its use; that is, protection from corrosion products was lost very rapidly because of the deterative properties of this material.

The inhibitor comparison data given in Table 6 were obtained with a new set of capillaries and also starting with a new set of Admiralty strips. As can be seen, the control runs gave considerably more corrosion than did those cited in Tables 4 and 5. Here again inhibitor I showed excellent effectiveness in reducing corrosion.

In an interesting series of papers, Dewar, et al.⁴ reported the use of a series of divalent sulfur compounds to promote dropwise condensation of steam on copper containing surfaces. This effect was ascribed to the formation of a tenacious film on the surface by the divalent sulfur compounds. However, such a film did not appear satisfactory to retard corrosion to a very high degree under the conditions which prevail in this test (Table 6).

TABLE 6—Corrosion with Admiralty Metal (New Strips) *

Name of Inhibitor	Wt Percent in Solution	Flowing Over Strip, ppm	Loss, mg
None	0	0	219, 286, 331; Ave. 279
Inhibitor I	0.10	20	56.54; Ave. 55
Lauryl mercaptan	0.10	20	109
Lauryl mercaptan	0.50	100	157
S ₁₀ (C ₁₈ H ₃₇ C-SCH ₂) ₂	0.10	20	203

* H₂S 35 cc./min; NH₃ 8.3 cc./min. Inhibitor Added at 0.5 cc./min. 20 hr. run.

TABLE 7—Inconsistencies in MIL-I-25017 ASTM Ratings of Coupons (Inhibitor I)

Inhibitor Conc, ppm	TEST NUMBER				
	1	2	3	4	5
4	Severe	Severe	Severe	Severe	Severe
6	Moderate	Moderate	Severe	Severe	Severe
8	Severe	Moderate	Severe	Severe	Severe
10	Severe	Moderate	Severe	Severe	Severe
12	Severe	Severe	Moderate	Clean	Clean
14	Severe	Severe	Severe	Severe	Severe
16					Severe
18					Moderate
20					Clean
22					Severe

TABLE 8—Testing on Coupons Segregated According to Meyer and Sheldahl⁵ (ASTM Ratings of Coupons)

Inhibitor Conc, ppm	Test No. Type of Paper and Meyer and Sheldahl ⁵ Coupon Group				
	1	2	3	4	5
	Garnet	Si C	Si C	Garnet	Si C
	Cor	Cor	Cor	Non Cor	Non Cor
20	Severe			Severe	
25	Severe			Severe	
30	Severe			Clean	Severe
40	Moderate	Severe		Clean	Moderate
50	Clean	Moderate		Clean	Moderate
60	Clean	Moderate		Clean	Moderate
70		Moderate			Clean
80		Moderate			Severe
100			Clean		
120			Moderate		
140			Moderate		
160			Moderate		
180			Clean		
"Min. Effect. Conc.", ppm	50	> 80	180	30	> 80

TABLE 9—Corrosion Inhibitor Testing by Spec. MIL-I-25017*

Additive	ppm (In Isooctane)	Micro-Inches Corrosion After 20 Hours Exposure		
		Run No. 1	Run No. 2	Run No. 3
None		67
Inhibitor G	5	65
	10	55
	20	50
	30	45	13	13
	35	..	4	1
	40	2	1	2
None		> 100
Inhibitor CC	5	83
	10	63
	20	54
	30	80	32	33
	35	..	31	26
	40	24	27	10
None		> 100
Inhibitor DD	5	> 100
	10	85
	20	23
	30	18
	40	3
None		> 100
Inhibitor L	5	> 100
	10	85
	20	53
	30	24
	40	18
None		> 100
Inhibitor I	5	> 100
	10	58
	20	3
	30	1
	40	0

* Modified to use resistance probes of SAE No. 1020 steel.

ASTM Turbine Oil Test D-665 Modified According to MIL-I-25017

It has been previously noted that the Turbine Oil Test does not show the effectiveness of a number of materials which appear effective by the test described above. A number of difficulties were encountered in carrying out these tests on the Turbine Oil apparatus. The following section discusses these points and describes a further modification of the tests.

The major objection to this method is the inconsistency of the results and the failure to obtain reproducible data. Thus, in a series of tests in which a corrosion inhibitor was used at concentrations of 10, 20, 30 ppm, etc. it was a frequent experience to obtain less corrosion at low inhibitor content than at higher content. Subsequent attempts to improve the reproducibility of this method were unsuccessful (Table 7).

Other investigators have had similar difficulties, as can be noted from the work of Meyer and Sheldahl⁵ in evaluating the ASTM Turbine Oil Test. This work showed that the corrosion coupons may be separated into two different corrosion groups by metallographic examination or by their relative rates of corrosion in Chicago tap water. Reproducible results were obtained using the more readily corrodible group of coupons and by polishing with silicon carbide rather than garnet paper.

Confirmation that there were at least two different types of corrosion coupons which fell into entirely different categories has been established. It was possible to separate these two categories in corrosion tests made with Lake Michigan water using the Meyer and Sheldahl technique. However, continuing to use this technique, wherein silicon carbide was substituted for garnet paper, the results were inconsistent and the amount of corrosion inhibitor necessary to inhibit effectively was very high, as shown in Table 8.

The use of garnet paper on the coupons selected in the manner described gave somewhat lower inhibitor requirement than the use of the silicon carbide, but the results were considerably higher than would be expected from the minimum effective concentrations approved by the military specifications for these inhibitors. The silicon carbide gave markedly deeper scratches than did the garnet paper; this difference was probably partially re-

sponsible for the greater corrosion of specimens polished with silicon carbide. The difficulties of trying to determine a "minimum effective concentration" are demonstrated in Table 8.

Actual rating of the corrosion coupons from the ASTM test is a subjective matter. MIL specifications require not more than six rust specks and if carried to the extent of looking for rust specks with a low-power magnifying glass, coupons which appear clear frequently can be observed to contain a number of specks. The recent appearance of a quantitative type of corrosion measurement involving change in conductivity of the corroded specimen as described in publications of Marsh and Schaschl² seemed to offer a possibility for obtaining more selective ratings of material. Accordingly, probes as described in their publication were modified so they

could be immersed in the ASTM Turbine Oil Corrosion beaker in place of the coupon, and readings were taken at intervals during a 20-hour test period.

Results of testing some corrosion inhibitors are shown in Figures 2 to 5. The data shown here are quite consistent and up to the present there has been only one instance of reversal. The difference in degree of effectiveness of the various corrosion inhibitors can be demonstrated readily by examination of the family of curves as shown in these figures.

It seems to be advantageous to determine a minimum effective concentration by ascertaining the quantity of inhibitor required to prevent corrosion in excess of an agreed number of microinches in a given time, rather than to rely on a subjective rating which is susceptible to variations with different operators. Additional

data obtained by this testing method are shown in Table 9. It can be seen in this tabulation that once the threshold where low corrosion prevails is attained, the results are reproducible. Above this threshold, where corrosion is still substantial, the measurement of extent of corrosion is less precise.

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Any discussion of this article not published above will appear in the December, 1959 issue

Topic of the Month

Effect of Velocity on Life Expectancy of Steel Pipelines In Commercial Strengths of Sulfuric Acid

By T. F. DEGNAN*

PIPELINES of carbon steel are the type most commonly used and generally most economical for handling commercial strengths of oleum and sulfuric acid above 78 percent. The corrosion resistance of steel depends, however, upon the formation of an insoluble protective film of ferrous sulfate. Velocity accelerates corrosion by removing the protective coating more rapidly than it forms. The grooving resulting from excessive velocity (11.9 ft per second) is shown in Figure 1.

An appreciation of the effect of velocity on the life expectancy of S/80 steel pipelines handling acid in the range 93-99 percent at ambient temperatures

may be gained by examination of data shown in Table 1. Service life was obtained from plant records.

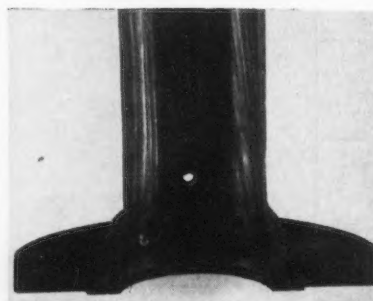


Figure 1—Failure of steel pipe after one year of service. Pipe handled 99 percent sulfuric acid at 11.9 feet per second at ambient temperature.

TABLE 1—Service Life of Acid Lines in Daily Service At Ambient Temperature

Acid Strength (percent)	Velocity (Ft/Sec)	Service Life (Years)
93.....	0.4	10
98.....	1.0	15
93.....	3.5	5
93.....	5.5	5-8*
93.....	6.5	3-5
98.....	6.7	1½
99.....	11.9	1

* Intermittent service, cast iron fittings.

* Engineering Service Division, E. I. du Pont de Nemours and Company (Inc.), Wilmington, Delaware.

Corrosion of Die Casting Alloys in Detergent Solutions Measured by Electrical Resistance Method*

By H. J. SMITH and R. L. HADLEY

Introduction

IN THE AREA of appliance performance the corrosion of metallic components can be a drastically limiting factor. Designing for adequate corrosion resistance is hampered by the lack of useful quantitative or comparative data concerning the resistance of commonly used metals to tap water, soap or detergent solutions and contaminated mixtures of both.

Under such conditions appliance designers have three choices:

1. To design on the basis of extrapolations of field service experience.
2. To rely on corrosion data for the closest approximate material and environment.
3. Deliberately to over-design with consequent high material cost.

The present investigation was undertaken with these needs in mind, utilizing a sensitive method of measuring small amounts of corrosion to obtain useful comparative data.

A survey of recent developments in the techniques of corrosion testing led to investigation of the advantages of electrical resistance methods for measuring the corrosion resistance of a metal specimen. These methods are based on two principles:

1. Metals and alloys generally have much lower specific resistance than their corrosion products.
2. Decrease in thickness of a metal specimen as a result of corrosion may be measured by measuring the increased electrical resistance of the specimen.

Dravnieks and Cataldi¹ utilized such a method for evaluating the corrosivity of crude oil mixtures employing thin steel shim stock (.001 inch) as specimens. Roller² employed a similar method for evaluating inhibitors for metallic containers for red, fuming nitric acid and for surface coatings on magnesium alloys. Lewis and Terrell³ used a resistance method for determining the corrosivity of naphtha streams in refinery processes. Marsh and Schaschl⁴ adapted a similar method to the rapid screening of corrosion inhibitors. All of these workers utilized thin metal ribbons from .001 inch to .020 inch thick and approximately .125 inch wide, varying in length from three to ten inches. At the present time at least two sources manufacture



Smith

Hadley

H. J. SMITH since 1955 has been with General Electric Company's Metallurgy and Ceramics Laboratory, Major Appliance and TV Receiver Division, Appliance Park, Louisville, Ky. For six years he was employed as a chemist and metallurgist at Fairmont Aluminum Co. in West Virginia. He later worked as a metallurgist at Tube Turns Plant No. 2 in Louisville and still later served as Assistant Works Metallurgist of International Harvester's Louisville Works. Mr. Smith is chairman of NACE's Ohio Valley Section and past chairman of ASM's Louisville Chapter. He attended Fairmont State College (AB degree, 1937) and West Virginia University.

R. L. HADLEY is Engineering Manager of General Electric Co's Metallurgical and Ceramic Development Group, Major Appliance Laboratory, Louisville, Ky. He received a BS Met. E. from Purdue University in 1942, and MS and PhD degrees from Carnegie Institute of Technology. Dr. Hadley was employed from 1949 to 1954 as research associate at General Electric Research Laboratory, The Knolls, Schenectady, N. Y. During World War II he served for four years in the U. S. Army.

and sell units for corrosion measurement employing this principle.

Conclusions drawn from this prior work showed the following advantages of the method:

1. The specimen did not have to be removed from the corrosive environment during the test.
2. Metal cleaning and weight-difference determinations, with their inherent susceptibility to error were eliminated.
3. Instrumental recording of corrosion during the test was entirely feasible.

Probable disadvantages appeared to be:

1. Temperature control and measuring techniques required design so results would not be affected by the temperature coefficient of resistance of the material.
2. Change in resistance and loss in weight do not bear a linear relationship through the entire course of the corrosion process.

Abstract

An electrical resistance method was adapted to test both aluminum and zinc die cast materials in tap water, detergent solutions, and detergent solutions plus bleach. Since the rate of attack of these materials in aqueous media is sometimes low, with consequent small resistance changes, low resistance circuitry is necessary. Plotted data show comparative corrosion resistance of zinc and aluminum materials and establishes the technique satisfactory for the intended use. Conclusions reached are: (1.) In all three test media the aluminum-silicon alloy No. 13 and the aluminum-silicon-magnesium alloy No. A360 are superior to the aluminum-copper-silicon alloy (A380) and the zinc die-cast material (D3). (2.) In solutions of detergent (not chlorinated) a reasonable amount of inhibition is conferred by the silicates which are present. (3.) No. 13 (aluminum-silicon) alloy appears to offer the maximum resistance to corrosion and No. 360 (aluminum-silicon-magnesium) alloy the next best. (4.) The method offers a satisfactory laboratory means of comparing corrosion resistance of metals. 2.3.5

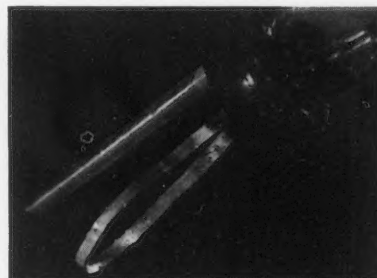


Figure 1—Ribbon specimen with electrical lead wires attached. Specimen, lead wires and glass tubes are sealed in rubber stopper with epoxy resin.

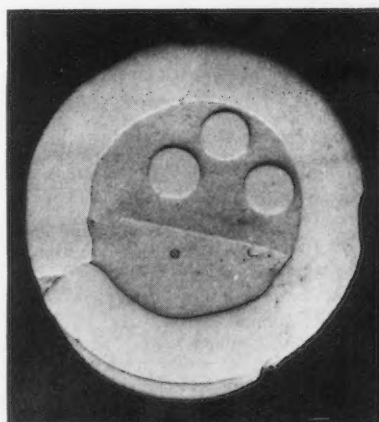


Figure 2—Cross-section of specimen-lead wire connection. Annular ring is copper connector, smaller circular sections are lead wire ends, and rectangle is specimen end. Matrix material is solder.

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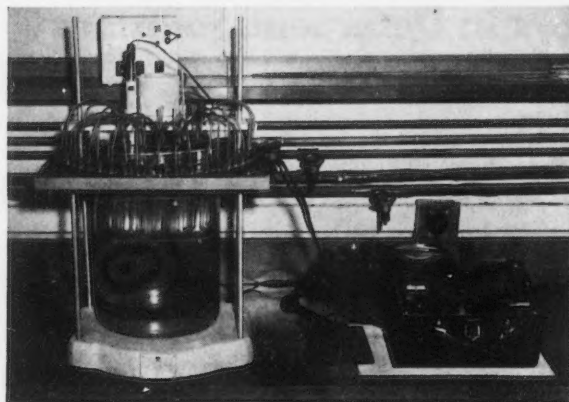


Figure 3—Test equipment used on corrosion testing of die cast materials. Constant temperature bath on the left contains specimens in tubes of corrosive media. Portable double bridge for measuring resistance is shown on the right.

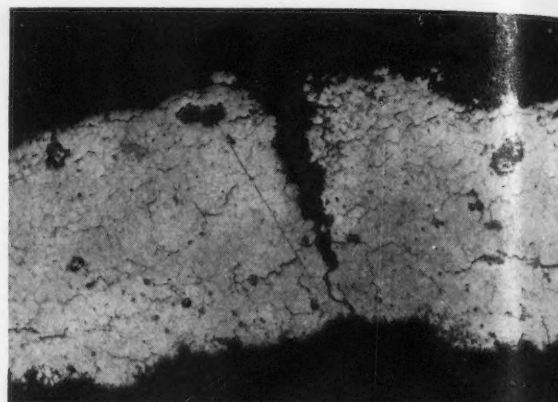


Figure 4—Specimen of Zn die cast material (D3) after 504 hours in a chlorinated detergent solution at 71 C. 200X.

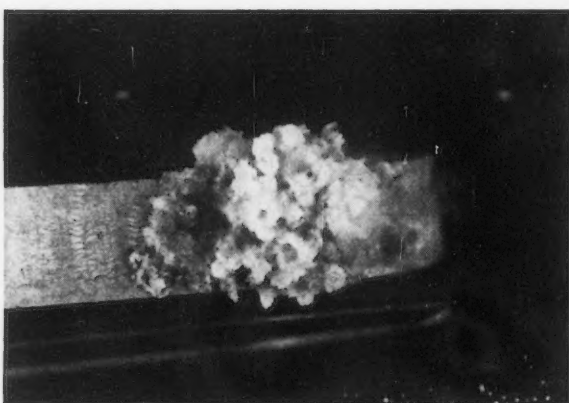


Figure 5—Products of corrosion in place on an A360 aluminum alloy specimen after 648 hours in chlorinated detergent solution at 71 C.

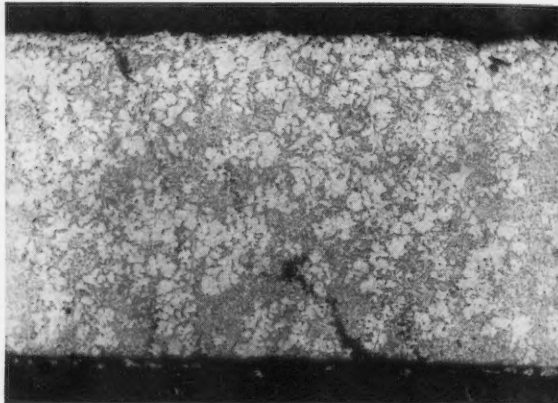


Figure 6—Photomicrograph of same specimen as shown in Figure 5. 200X.

TABLE 1—Louisville (Ky) Tap Water

Total Dissolved Solids.....	161.6 ppm
Total Hardness (CaCO ₃).....	102.4 ppm
Alkalinity as CaCO ₃	42.0 ppm
Free CO ₂	0.9 ppm
Manganese.....	None
Iron.....	0.8 ppm
Copper.....	0.07 ppm
Chlorides (Cl).....	17.7 ppm
Sulfates (SO ₄).....	46.8 ppm
Nitrates.....	0.8 ppm
pH.....	7.7

It was readily apparent that previously reported techniques in this field would need to be modified to meet the following conditions:

1. The electrical resistance of the zinc and aluminum materials under consideration are lower than steel by factors of $2\frac{1}{4}$ and $3\frac{3}{4}$ respectively.
2. The reaction rates of zinc and aluminum in many waters and detergent solutions are appreciably lower than steel in an acidic solution.
3. Die-cast materials would need to be prepared in ribbon form.
4. Due to the low total circuit resistance involved usual electrical connectors might be unsatisfactory.

TABLE 2—Die Casting Alloys (Chemical Composition)

Element	CONTENT OF ALLOYS, PERCENT			
	*SI2A No. 13	*SC84A A380	*SG100A A360	*AG40A D3
Si.....	11.26	8.73	9.22
Al.....	Bal	Bal	Bal	3.95
Cu.....	0.39	2.20	None	0.05
Mg.....	0.12	0.05	0.53	0.28
Fe.....	0.03	0.65	0.51	Trace
Mn.....	0.18	0.32	0.03
Zn.....	0.15	0.11	0.08	Bal
Ni.....	None	0.24	0.05
Sn.....	0.04	0.03	0.03	None
Cd.....	None
Pb.....	None

*ASTM Designation.

Specimen Preparation

Specimens of the various die-cast materials were cut from $\frac{1}{8}$ inch thick die-cast slabs (4 inches x 6 inches) furnished by commercial die casting suppliers. Samples of each material were taken for chemical analysis. Strips 3 inches wide and 6 inches long cut from the original slab were lightly sanded on each face, degreased and cemented to ground steel blocks using an epoxy adhesive. Clamping pressures were relatively light and the adhesive (catalyzed) was cured by standing at room temperature for a pe-

riod of 24 hours. After curing a milling operation was employed to reduce the original $\frac{1}{8}$ inch thickness to .015 inch-.020 inch. Milling tool marks were removed by polishing with fine abrasive paper. The assembly was then heated to 300 F which softened the adhesive and the thin sample of cast material was peeled from its steel block.

The specimen then was bent to a U shape, copper lead wires were attached and the assembly carefully degreased with organic solvents. Chemical cleaning prior to testing was not employed be-

cause of the porosity and interconnected voids which would trap the cleaning material. The specimen thus prepared furnished two surfaces for simultaneous testing, one essentially as-cast (tension side of bend) and one as-machined (compression side of bend). A typical specimen is shown in Figure 1.

Test Equipment

The looped ribbon specimen was sealed into a rubber stopper with an epoxy resin and was then inserted into a 200 ml borosilicate test tube filled with the appropriate test solution. Waterproof tape was used to ensure against loosening of the stopper in the tube. Two $\frac{1}{8}$ inch glass tubes were also inserted through the stopper to allow filling and draining of the test tube if desired. The glass tubes projected three inches above the stopper and served as a "standpipe" to allow a higher liquid level and eliminate an air-liquid interface on the test specimen.

Electrical connection was made between specimen and lead wire (No. 12 stranded copper) by compressing a soft solder mechanically, without heat between the wire, specimen end, and an annular connector. Figure 2 shows a cross section of a typical specimen connection.

The other end of the lead wires were submerged in small, individual glass tubes of mercury which served as stable, low-resistance connectors. When measurements were made leads from the test instrument were submerged in the mercury tubes and readings were taken.

The measuring instrument employed was a G. E. portable double bridge with a range of 22 ohms to .0001 ohm. A magnifier was utilized to detect the slightest deviation of the galvanometer pointer.

Individual sample tubes were supported on a rack immersed in an oil-filled constant temperature bath capable of maintaining temperatures within $\pm .1$ C.

A general view of the test equipment is shown in Figure 3.

Test Procedures

Duplicate samples of test material were utilized and samples of a wrought aluminum material (1100) were tested for comparative purposes.

The corrosive media used were Louisville tap water (composition shown in Table 1), a 0.38 percent solution of a standard detergent, and the same detergent solution +0.4 percent of a common liquid household bleaching agent. The samples were maintained at a temperature of 71 C and the solutions were not changed for the duration of the test.

In early tests a standard sample of 1100 aluminum strip (.010 inch x .125 inch x 6 inches) was immersed in an inert oil-filled tube and was held in the bath with daily readings taken as a check against thermal, contact, and electrical measurement variations. Triplicate readings on specimens were made daily and a plot of resistance vs time was

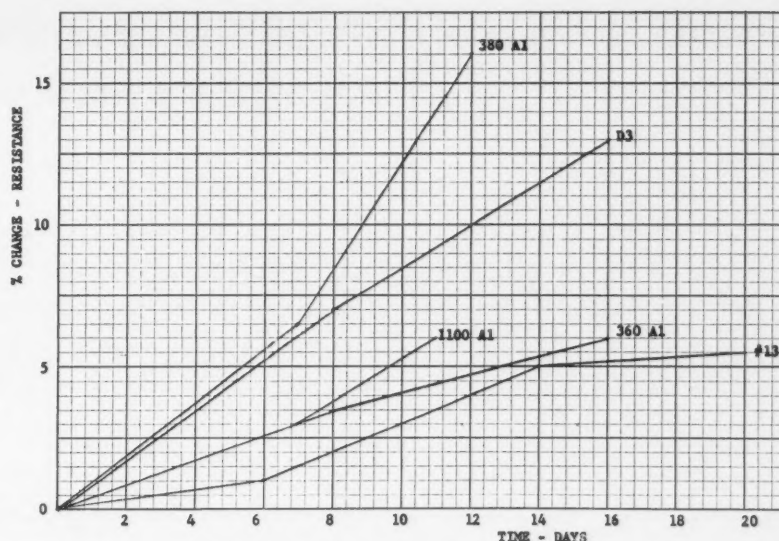


Figure 7—Test results in tap water (stagnant) at 71 C.

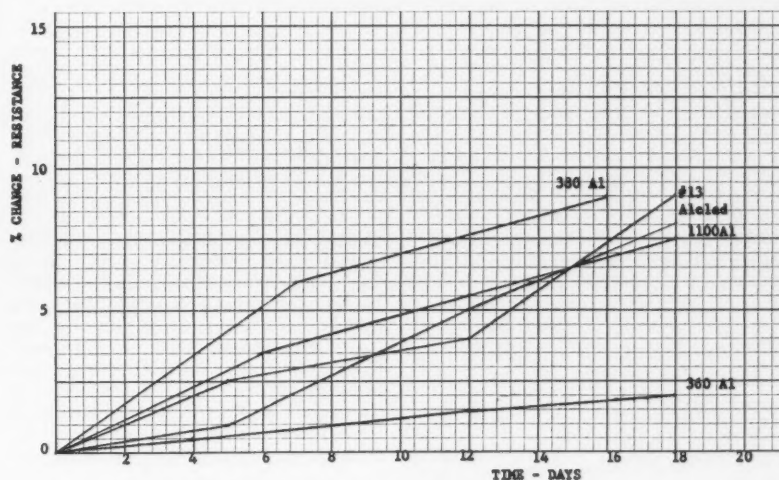


Figure 8—Test results in detergent solution (stagnant) at 71 C.

maintained until resistance increased abruptly or actual failure occurred. The cast materials used and their chemical composition are shown in Table 2.

Discussion of Results

It was observed that the attack of zinc material was of a general nature while the aluminum specimens tended to corrode at a few discrete sites. A corroded test specimen of D3 material is shown in Figure 4. The corrosion products evident on a specimen of A360 aluminum alloy are shown in Figure 5. A photomicrograph of the same specimen is shown in Figure 6. Comparing the cast aluminum material with the wrought (1100) standards it was noted that the cast material was more susceptible to intergranular attack and more localized corrosion. This was attributed to the greater heterogeneity of the cast material with impurities in large grain boundary areas. In tests run to date there ap-

peared to be no tendency for either the machined or as-cast side to corrode preferentially.

With samples of .015 inch x .125 inch cross section a 10 percent resistance change would mean a reduction of 1.6 mils in thickness or, assuming a uniform loss, a removal of .0008 inch from each surface (discounting edges). The sensitivity of the method is such that an aluminum sample with an original resistance of .01225 ohms can have changes measured as low as 0.4 percent or a resistance change of .0005 ohm. This would correspond to a metal loss of about three millionths of an inch per face. The reproducibility appears to be satisfactory within the expected limits of the heterogeneous material being tested. It would seem that even very localized pitting attack can be satisfactorily measured within some limit of pitting frequency and distribution not yet determined.

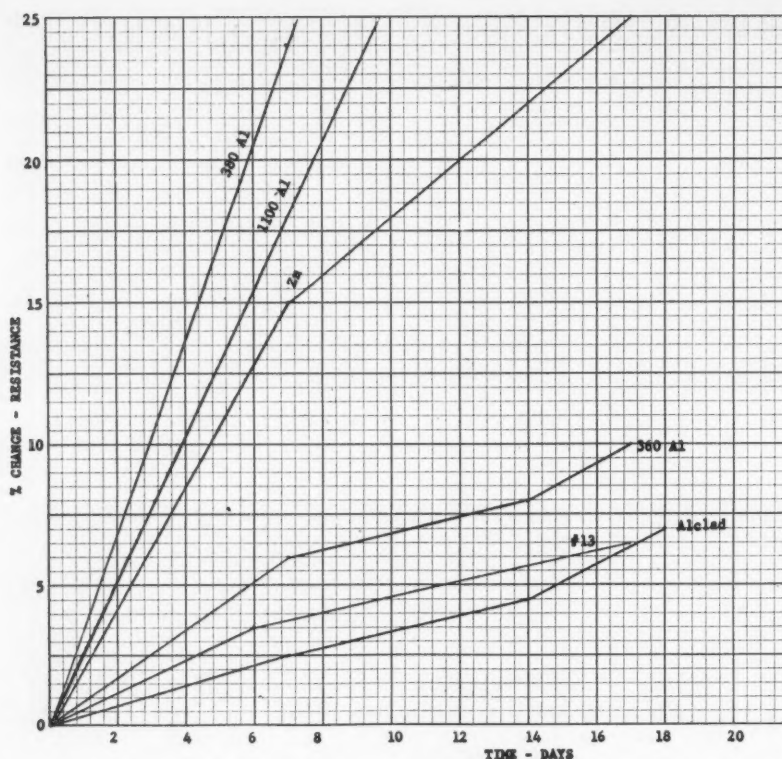


Figure 9—Test results in detergent plus bleach (stagnant) at 71 C.

The detergent solution was comprised of a standard commercial detergent with a nominal composition as follows:

Material	Percent
Organic surfactant	13.0
Sodium carbonate	22.2
Polyphosphate	40.0
Sodium silicate	24.0
Anti-redeposition agent	0.8
Optical bleach	0.1 (max)

A 0.38 percent solution of such a detergent shows a pH value of 10.0 at the test temperature. The active ingredient of the bleach is sodium hypochlorite with about a 5 percent active chlorine content. The pH of this material ranges from 10-11. The relation of specimen area to volume of test solution was approximately 150 ml of solution per sq inch of specimen.

Reference was also made to work done

regarding the influence of specimen area on pitting probability by Aziz and Godard.⁵ While variation will be encountered in different media, the specimen size employed is approximately the size beyond which Aziz and Godard found no increase in pitting probability.

Data for three series of tests, each in a different solution, are shown in Figures 7, 8, and 9.

Conclusions

1. In all three test media the aluminum-silicon alloy No. 13 and the aluminum-silicon-magnesium alloy No. A360 are superior to the aluminum-copper-silicon alloy (A380) and the zinc die-cast material (D3).
2. With all of the materials tested the tap-water was more aggressive than the non-chlorinated detergent solution.
3. No. 13 (aluminum-silicon) alloy appears to offer the maximum resistance to corrosion and A360 (aluminum-silicon-magnesium) alloy the next best. (These findings are in partial agreement with Colwell and Kissling⁶ who used alternate immersion and measured reduced mechanical properties. They found No. 13 alloy to be most resistant but found A380 alloy to be superior to A360).

A correlation of data from this work with available field service data should greatly improve the ability to predict the performance of metals in appliances.

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Progress Report on Inhibiting the Corrosion of Steel In a Reinforced Concrete Bridge*

By R. F. STRATFULL*

Introduction

GALVANIC CORROSION of the reinforcing steel in the 7-mile San Mateo-Hayward Bridge, which crosses the southern arm of the San Francisco Bay, has been a subject of study for several years. Two papers describing the causes of corrosion of the reinforcing steel in this structure have been published.^{1,2}

This paper is a progress report on three experimental techniques employed in an attempt to inhibit corrosion. The first trial method of corrosion control is cathodic protection, the second is oxygen starvation by adding capillary water to the concrete and the third is moisture equalization within the concrete by painting a moisture-resistant membrane over the surface of the cathodic areas of the deck units.

Repair of Corrosion Damage

Magnitude of the corrosion problem on this 30-year-old reinforced concrete structure can be illustrated by the fact that the State of California spent almost two million dollars within the last decade for repairs caused by corrosion damage.

Visible effects of corrosion of the reinforcing steel are similar to the behavior of other reinforced concrete structures showing distress. The concrete is cracked or spalled. At times there are rust stains on the concrete surface. When the steel is examined, it may have thick stratified onion skin rust on its surface. Usually the steel is pitted.

Repair of the reinforced beams containing corroded steel is accomplished by orthodox methods: the concrete is chipped from the effected areas, the steel is sandblasted and then the girder is rebuilt using cement mortar shotcrete with wire mesh reinforcing to complete the repair. If the steel stirrups are reduced measurably in area, they are repaired by replacing the damaged area.

After these repairs are completed on the damaged portions of a particular span, it might seem that no further trouble should be encountered. However, corrosion often reappears elsewhere in the same span, and this difficulty is of sufficient magnitude to cause concern.

Causes of Corrosion

Two basic environmental factors seem to cause the macro-galvanic corrosion of the reinforcing steel in this bridge: ab-

sorbed moisture and absorbed sea salts. These factors appear to be interrelated.

Concrete will become an electrolyte when moisture is present.³ When moisture content of the concrete is equal, corrosion cells will be formed by differential salt content.^{1,2,3} When the salt content is equal, differential moisture contents can cause the steel to become anodic or cathodic. A galvanic couple of steel in moist concrete will become anodic to steel in relatively dry concrete.^{1,2}

In addition to the fact that moisture and salt content in the concrete influences the presence of the corrosion cell, it was found that, when an anodic area of steel was repaired by shotcrete, this previously anodic area became cathodic with respect to undisturbed steel embedded in the original concrete. Thus, when the concrete damaged by corrosion of steel is repaired in one area, the attack is diverted to a new area.

These causes of corrosion have been amplified in laboratory and field tests covering the corrosion of steel in concrete.^{1,2} The role of sea salts in promoting the corrosion of reinforcing steel in electrolytes of moist concrete has also been confirmed.³

Macro-Galvanic Corrosion Cell

General arrangement of the macro-galvanic cell, which contributes to bridge's corrosion, has anodic and cathodic areas separated by distances of 2 to 10 feet in the reinforced concrete deck slab. Generally, the deck is cathodic to the beams. The caps and the piling have cathodic and anodic areas which may be in random locations. Potential measurements made on the surface of the structure with a copper sulfate half-cell readily disclosed the potential distribution pattern and the location of anodic and cathodic areas.

Similar potential measurements showed that the same type of galvanic corrosion occurs in abandoned pilings in salt water environment and in laboratory test cells^{1,2,3} wherein the anodic and cathodic electrodes were separated by distances as small as a few inches or as great as 10 feet.

Because of the size and distribution of the macro-galvanic corrosion cells in the San Mateo-Haywood Bridge, the over-all damage poses a difficult problem. However, the present rapid rate of corrosion may diminish with time. This factor is brought out by potential measurements conducted upon the concrete surface in spans which have been repaired. Care-

Abstract

Experimental techniques used to inhibit the corrosion of steel in a reinforced concrete bridge are described. Control methods used include application of cathodic protection, addition of capillary water to the concrete to produce oxygen starvation and use of paints to produce moisture equalization within the concrete. Role of absorbed moisture and sea salts in promoting corrosion is discussed briefly. Because several tests still remain to be made and evaluated, data are presented as a progress report, not as conclusions for design criteria. 5.2.1

fully repaired beams which were once anodic to the deck area prior to the repair often become cathodic after the repair. This polarity reversal causes corrosion to shift to the deck reinforcing steel. However, because the under deck has a smaller concentration of salt and moisture, the rate of corrosion is anticipated to be less than that of the beam sections. This factor of sea salt concentration and penetration into the structure has been evaluated by electrical resistivity and potential measurements of the concrete and steel in the field and by laboratory sampling of core-drilled specimens.^{1,2}

Fundamental corrosion problems in this bridge are not unique; Halstead and Woodworth were among the first to study similar phenomena in Natal, South Africa. Also, preliminary investigation of a reinforced concrete bridge adjacent to the surf near San Diego, California, indicates that the structure's reinforcing steel was suffering from similar macro-galvanic corrosion.

Cathodic Protection for Bridge Structure

Cathodic protection of metal buried in soils or immersed in water is a commonplace expedient. However, the exposed or aerial application of cathodic protection to a reinforced concrete bridge beam and deck unit which is well above water level appears to be unique. Yet, if the moist concrete can function as an electrolyte, then cathodic protection should be possible.

Anode Assembly

For experimental purposes, a one-sided wooden trough or retainer was made of Douglas fir which had been pressure treated with pentachlorophenol preservative. This trough was attached by its exposed side to the perpendicular face of the reinforced concrete beams to hold the anode assembly. Figure 1 shows the anode trough assemblies mounted in place on the bridge structure.

For an anode, a 3-inch x 60-inch carbon rod was fully embedded in the anode trough. To insure electrical contact be-

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*Corrosion Engineer, California Division of Highways.

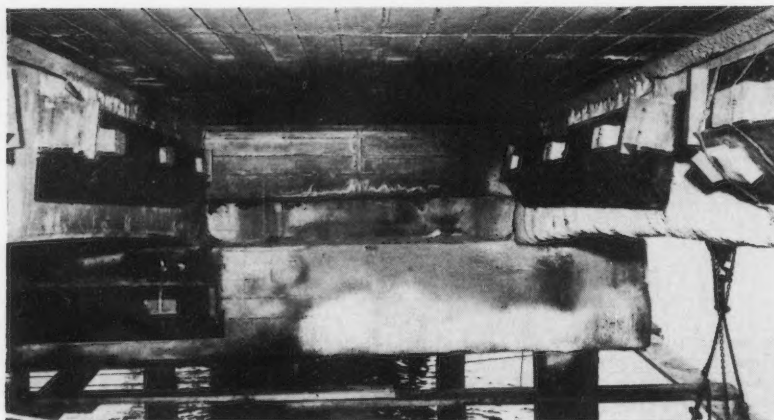


Figure 1—Experimental anode assemblies placed on sides of bridge beams and caps for application of cathode protection.

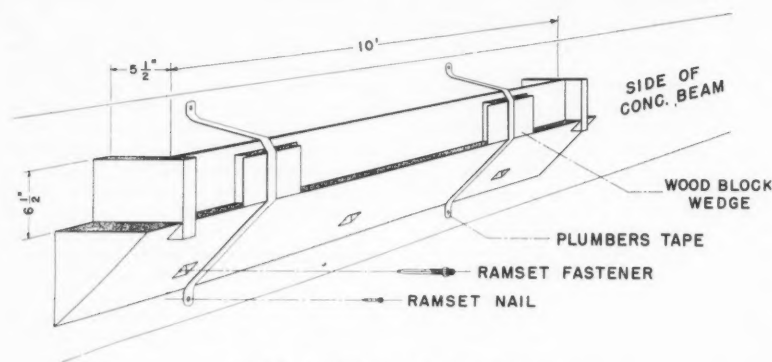


Figure 2—Detail drawing of anode assembly.

tween the anode and the surface of the concrete and to maintain good current distribution from the anode, an electrolytic backfill was placed around the carbon anode. This backfill consisted of a slurry of 1 part raw gypsum to 4 parts steam exploded mica by volume. To the total volume of raw gypsum and mica, approximately 10 percent by volume calcium chloride was added.

After the paste-like electrolyte was placed in the anode trough, approximately 0.20 cubic feet of calcium chloride was sprinkled on top of the electrolyte. Measurements made after a 3-month field exposure showed the resistivity of the electrolyte to be 75 ohm cm. Figure 2, a detail drawing of the anode assembly, shows the dimensions and method of constructing the unit.

Applied Voltage for Cathodic Protection System

Because cathodic protection of reinforcing steel in a concrete structure is relatively new, no information is available regarding the voltages which should be employed. Extensive experiments were conducted to determine the minimum applied voltage which would give the greatest protection over the structure with the least number of anodes. Reports in the literature^{4,5,6,7} refer to the deterioration of the bond between reinforcing

steel and concrete when high voltages are applied. However, unpublished data by J. L. Rohwedder and F. W. Shanks of the U. S. Army Corps of Engineers cites the loss and eventual regain of the bond strength of cathodic steel under various current densities ranging from 2 to 50 ma per square foot.

While establishing a voltage requirement for this application of cathodic protection, one of the experiments was conducted to determine the total electrical resistance of the complete cathodic protection circuit from the experimental carbon anode to the reinforcing steel cathode.

Results of the circuit resistance measurements are shown in Figure 3, a plot of circuit resistance versus applied voltage. The total circuit resistance does not materially change when the d-c voltage exceeds approximately 3 volts, particularly when compared to a smaller voltage. The d-c circuit resistance of approximately 30 ohms at 8 volts d-c compares closely to the circuit resistance measuring instruments. The apparent change in circuit resistance with various applied voltages was also detected in measurements of laboratory test cells which consisted of two steel electrodes embedded in concrete.

As indicated by the chart in Figure 3, a minimum of 3 volts should be applied

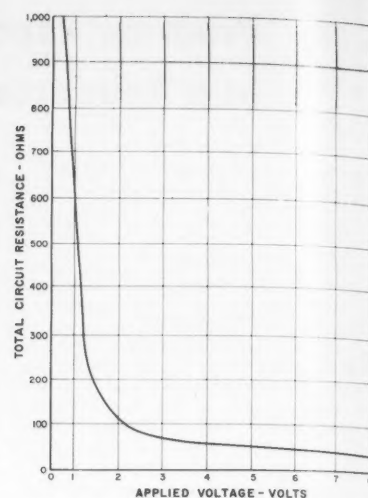


Figure 3—Plot of circuit resistance versus applied voltage.

to this installation for the apparent economic application of cathodic protection currents.

Potential Change and Applied Voltage

In conjunction with the economics of circuit resistance and applied voltage for the cathodic protection of reinforcing steel, consideration must be given to the magnitude of the applied voltage for corrosion inhibition.

After a search of the literature did not disclose sufficient data indicating the numerical value of the protected potential of steel embedded in concrete, no attempt was made to cathodically protect the steel from micro-galvanic corrosion. The criteria for the protection of the steel was assumed to be the most economical distribution of cathodic protection currents which would remove or decrease those anodic areas about the reinforced steel structure where there was definite evidence of macro-galvanic corrosion.

Evidence of the distribution pattern of cathodic protection currents was determined comparatively by first surveying and plotting the original potential of the steel to a copper sulfate half-cell; then the half-cell potential of the same reinforced steel structure was measured after cathodic protection was applied.

Survey of the potential of the steel after the application of cathodic protection (current-off measurements) was closely examined for evidence of stray current damage. In approximately 75 separate cathodic protection tests, there was no evidence, as indicated by potential measurements, of stray current damage to the reinforcing steel.

Because of the geometric configuration of the bridge and its mass of reinforcing steel, apparently there were no practical means by which the actual current density to the reinforcing steel could be measured or calculated directly. Therefore, calculations have been limited to the square feet of concrete surface which indicated a potential change of the reinforcing steel.

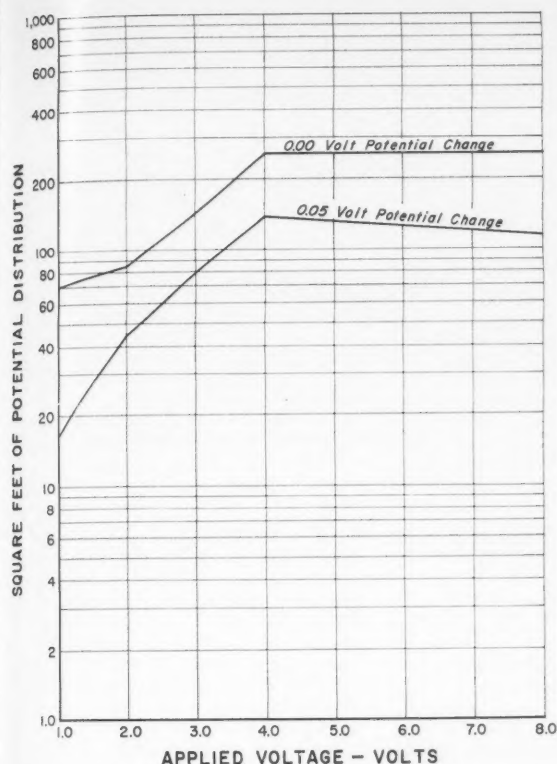


Figure 4—Plot of the area of potential distribution versus applied voltage.

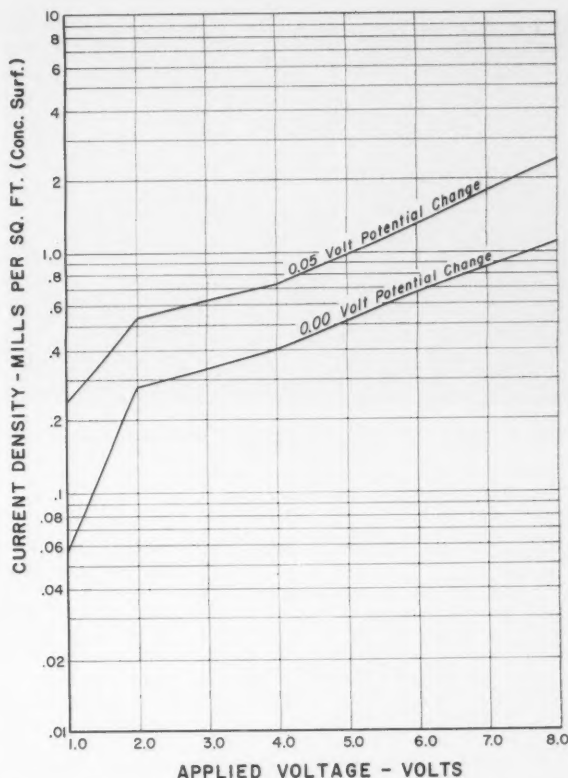


Figure 5—Plot of the average surface current density versus applied voltage.

Figure 4, a plot of the area of potential distribution versus applied voltage, indicates the distance of current throw across the surface of the structure when various voltages are applied. This chart is the result of plotting the differences between the potential of the reinforcing steel to concrete surface before and after cathodic protection and of measuring the enclosed area between the potential changes indicated.

As indicated in Figure 4, the actual surface area of concrete on which a potential change was measured did not increase materially after 4 volts were applied.

The change in the average current density on the surface of the concrete when subjected to an increase in applied voltage is shown in Figure 5, a plot of the average surface current density versus applied voltage.

As of June, 1958, experimental cathodic protection of the reinforcing steel in one span of the San Mateo-Hayward Bridge had been under test continually for 10 months. Thus far, potential measurements and field inspection indicate that the installation is operating satisfactorily. Satisfactory operation is judged by the lack of new anodic areas of evidence of stray current corrosion of the steel or spalling of the concrete.

Corrosion Control by Adding Capillary Water

In previous studies of the corrosion of reinforcing steel, concrete was found to have a higher electrical resistance in ca-

thodic areas than in the anodic areas. Also, the greatest influence on the electrical resistivity of concrete was created by changes in moisture content, particularly in the cathodic areas.^{1,2}

Laboratory experiments indicated that if a concrete test cell containing two steel electrodes was completely immersed in water (which excluded air from the voids) the galvanic currents due to differential salt contents generally would be reduced to insignificant values. Conversely, if a concrete cell of equal salt content was subject to differential wetting by moisture, galvanic corrosion currents were found to flow. The wetter element became anodic.

Therefore, to evaluate the phenomenon of differential moisture content as a method of corrosion control, one half of a span in the structure was wetted in the normally cathodic area by attaching wetting units to the underside of the deck. Figure 6 shows the wetting units attached to the underside of the deck. Figure 7 is a detail drawing of the construction and installation of the troughs used as wetting units.

A water reservoir (not shown) is attached to the outside of the bridge railing, and fresh water is piped by gravity to the wetting units. In addition to the wetting units, a 10-mil thickness of vinyl paint was placed on the underside of the deck, sides and shotcrete repaired areas of the beams. The locations on the bottoms of the beams where the potential measurements indicated the steel to be anodic were not coated with paint.

Observations for 6 months of the potential measurements in this experimental span do not indicate that any conclusions could be established for this installation. However, potential measurements in the span before application of moisture indicated that the ratio of cathodic to anodic potential areas (relative surface areas of concrete encompassed by anodic or cathodic potentials) on the surface of the concrete was altered from 15 units cathodic to 1 unit anodic to 4 units cathodic to 1 unit anodic area after application of moisture to the deck.

This corrosion control method may possibly reduce the corrosion rate since the ratio of the cathodic to the anodic potential areas has been reduced. Laboratory tests indicated that water-saturated cathodic areas stopped practically all galvanic action and currents. However, wetted cathodic areas on the bridge possibly are not being sufficiently saturated with moisture and, as an alternative to reducing galvanic activity, corrosion is being accelerated in the anodic areas. Thus, the advantages or disadvantages of the cathodic wetting technique may not be fully evaluated for several years.

Moisture Equalization by Painting

Moisture equalization in concrete cells in the laboratory usually reduced the flow of galvanic currents. Thus, as a corrosion control method, painting of the concrete underdeck, sides and beams (excluding the shotcrete repaired areas) was

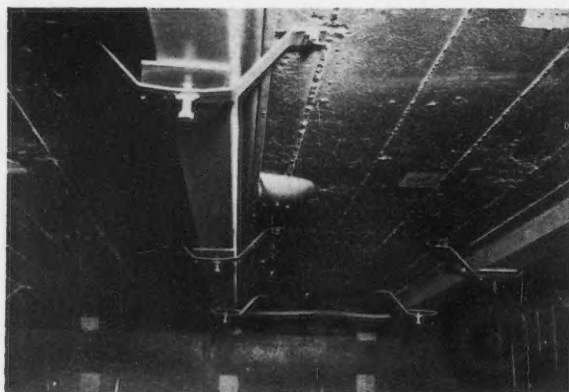


Figure 6—Wetting units attached to underside of bridge deck to add capillary water to the concrete.

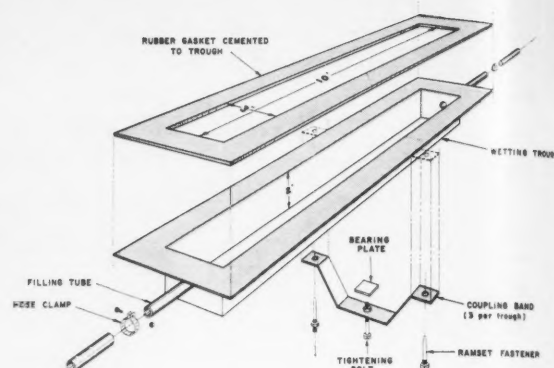


Figure 7—Detail drawing of the wetting trough used.

tried. A 10-mil thickness of vinyl paint was applied.

Purpose of the coating over the concrete was an attempt to reduce evaporation or absorption of moisture which occurs with each weather change. In theory, this moisture control would permit gradual equalization of moisture content throughout the entire unit.

In this bridge span, as in the other experimental spans, potential measurements were taken before paint application. After the span was painted, small areas of paint were stripped from various locations to facilitate subsequent potential measurements. To date, no definite conclusions can be drawn because of the test exposure of only 6 months. However, as a tentative indication, the ratio of cathodic to anodic potential areas are again compared: before application of the paint coating, the concrete surface area of the two potentials was 25 units cathodic to 1 unit anodic potential surface area; after coating application, the concrete surface area relationship of the potential of the steel changed to 67 units cathodic to 1 unit anodic surface potential area.

Apparently, the increase in the ratio of the cathodic to anodic areas by paint application may indicate that corrosion

is being localized and perhaps accelerated by this method of corrosion control.

Conclusions

In conclusion, it is emphasized that the data contained in this paper constitute a progress report and should not be accepted as conclusive for design criteria.

Numerous tests and measurements remain to be made if any of the experimental methods of corrosion control are to be evaluated.

This experimental corrosion control program will be continued. Also, new methods of corrosion control will be considered if they appear to be economical.

Acknowledgments

This investigation and trial installation of cathodic protection on reinforcing steel was conducted as one of the activities of the Materials and Research Department of the California Division of Highways.

The author wishes to express appreciation to F. N. Hveem, Materials and Research Engineer, J. L. Beaton, Supervising Highway Engineer, Bailey Tremper, Supervising Materials and Research Engineer, and L. S. Hannibal, Senior Mechanical Engineer of the Ma-

terials and Research Department for their invaluable advice and direction during this study. To H. C. Wood, Bridge Engineer, and M. W. Gewertz, Senior Bridge Engineer, for their aid and cooperation during this study. Also to Major F. W. Rhea of the U. S. Army Corps of Engineers who made available for reference the work of J. L. Rohwender and F. W. Shanks.

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Any discussion of this article not published above will appear in the December, 1959 issue



DISCUSSIONS

Development of the Redox Probe Field Technique by F. E. Costanzo and R. E. McVey. *Corrosion*, Vol. 14, No. 6, 268t-272t, (1958) June.

Comments by L. P. Sudrablin, Electro Rust-Proofing Co., Belleville, New Jersey:

The authors report pitting at Survey Stations 12517 + 21 and 12532 + 14 on the 10-inch bare pipeline which had magnesium anodes attached to it at the time of construction (shown in Figure 12). Since pitting can be the result of local non-uniformities in the soil contacting the pipe, have the authors been able to identify the presence of disseminated bits of clay, for example, in generally well aerated porous soils?

The discussor will caution against interpreting the pipe potentials measured to a Cu-CuSO₄ reference electrode of —.95 volt and —.90 volt, respectively, as being a significant criterion in the control of the short lines of corrosion current flow associated with localized pitting. The pipe potential measured to the reference electrode positioned on the ground surface over the pipeline includes an IR drop effect, a large part of which has no effect on the control of local cell corrosion. The IR drop through the soil, in the case reported, is attributable to the flow of protective current from the magnesium anodes. The significance of the reference electrode position in the control of local cell (pitting) corrosion has been demonstrated (L. P. Sudrablin and F. W. Ringer, *Corrosion*, 351t-357t (1957) May.)

Extending this discussion on the criteria used by the authors, others (B. McCollum, and K. H. Logan, National Bureau of Standards Technical Paper No. 351, 1927) have reported on the limitations of the earth current meter for corrosion systems having short lines of corrosion current flow, thusly: "... under favorable circumstances, it is possible to pick up, with a 4½ inch contactor, evidences of current flowing between a corroded and an uncorroded point on a section of a pipe, providing the path of this current is long enough to include both the potential terminals of the electrode. However, the contactor picks up but a component of this current and it is not possible to compute the true galvanic current from the reading obtained. If the galvanic circuit is very short, as is often the case, the earth current meter will be only slightly affected, if at all."

It is felt that more useful correlation between the redox potential technique and other criteria will be obtained by recognizing the limitations of each criterion used.

Reply by F. E. Costanzo:

In reference to the presence of disseminated bits of clay, we have not been able to identify any clay since the entire area in question is a peat bog, and a pipeline is generally saturated with moisture.

Samples of the black corrosion products were lifted from the pipe in pieces as large as your hand, and bright metal has been observed on the pipe. Samples of the corrosion products placed in dilute hydrochloric acid indicate the presence of sulfides, confirming the presence of sulfur-reducing bacteria.

In respect to the Cu-CuSO₄ reference electrode of —.95 volt and —.90 volt respectively, it is quite possible that the position of the electrode may account for this. However, the readings of the redox potential and the Cu-CuSO₄ potential were not at the exact same spot when the data were taken.

Since the earth current meter was presented by the National Bureau of Standards in 1927, considerable investigations have been made in evaluating the instrument for galvanic currents. We refer to some work reported at the Pittsburgh International Conference on Surface Reactions in 1948. The instrument was discussed in "Measurement of Galvanic Currents Around an Underground Structure" by N. P. Peifer and F. E. Costanzo.

We feel there is enough correlation of data with the earth current meter so that it can be used to correlate corrosion testing techniques.

Coatings for Underwater Metal Surfaces in Fresh Water Exposures by Sol M. Gleser. *Corrosion*, Vol. 14, No. 8, 337t-386t, (1958) August.

Comments by Warren D. Palmer, Protective Coating Engineering and Inspection Service, Houston, Texas:

It is my opinion that the technical formulation data presented in this paper in general is of little true meaning to those interested in corrosion mitigation or long life, lower maintenance cost. The findings and truths of the text has been general knowledge for about ten years to those engineers working in the protective coatings field.

The conclusion by Sol M. Gleser, "It is believed that it will be possible to accurately predict the behavior and characteristics of various coatings in various exposures. When that time arrives, painting will really have become a branch of the engineering sciences", is good reading but is already ten years late. In my opinion, such statements made in *Cor-*

rosion create the impression that protective coating engineering is not yet a true science; that it will be yet some time well in the future before sound and economic savings from protective coating predictions can be made of particular corrosion preventive application by engineers and personnel in the know. This is not true. There are already several engineers who for some time have been making most accurate and dependable protective coating performance predictions and economic saving evaluations.

The main problem is to get management, design and other engineering consultants to follow the protective coating engineer's recommendations, and to avail themselves of his service in time that the planning, selection and the proper application can be made to new construction.

It is not my intent to create the idea or impression to readers that the qualified protective coating engineers have all the answers or are stagnant in their search for new materials, methods of application, use, etc. to improve and up grade performance and lower corrosion mitigation cost for industry. However, the time is already at hand when protective coating engineering for corrosion control or reduced loss from corrosion is functioning with the same dependability as the other engineering sciences.

Reply by S. M. Gleser:

From my observations, it appears that, unfortunately, many authors reporting on performance of paint coatings fail to give precise quantitative information relative to formulation, environment, surface preparation, methods of application, performance of coating and the like. Instead they content themselves with broad generic designations and qualitative descriptions of performance. Terms such as "phenolic-modified tung-oil," "red lead and oil paint," "spar varnish" and the like are often used to describe coatings in lieu of the inclusion of their precise formulation. Similar imprecise language is used for the other elements of the coating system. It is obvious even to amateurs that there might be a great difference in performance in two tung-oil varnishes prepared with two different phenolic resins or even with different quantities of the same resin. Also, differences in performance can be expected between coatings having variations in any of the other elements of a coating system and its environment. To the extent that the literature of protective coatings fails to give precise data, to just that extent will the practices of protective coatings remain an art rather than a science. Similarly, unless precise data are made available in the literature, training of new corrosion engineers will

be a matter of apprenticeship rather than of scholastic endeavor.

The dictionary defines a science as "a branch of knowledge or study dealing with a body of facts or truths systematically arranged and showing the operation of general laws." Painting is only now beginning to reach that status as publications like *Corrosion* make available to the engineering profession precise data on performance which can be systematically arranged to show the operation of general laws. While it is true that there are a number of engineers capable of making more or less dependable predictions as to the performance of coatings in a new environment, it is also true that much of their knowledge is based on data which for the most part are not published and the precise details of which often are kept as trade secrets.

Mr. Palmer states "the time is already at hand when protective coating engineering for corrosion control or reduced loss from corrosion is functioning with the same dependability as the other engineering sciences." I know of no reputable corrosion engineer yet willing to state that he can predict the performance of a paint coating in a new environment with the same precision that a chemical engineer can predict the results of a reaction in a retort, a civil engineer the strength of beam, or a mechanical engineer the life of a bearing. There are records of failures of paint coatings applied in accordance with recommendations of reputable corrosion engineers, indicative of the still limited knowledge of the profession. The important thing is that the complete records concerning these failures be made available to the profession as well as those concerning coating successes, so that they can be used as the building blocks in deriving our concepts of the general laws governing the science of paint coatings.

Failure of Type 316 Stainless Steel Autoclave Components by J. P. Hugo and L. G. Nel. *Corrosion*, Vol. 14, No. 12, 553t-556t, (1958) Dec.

Comments by C. P. Dillon, Union Carbide Chemicals Company, Charleston, West Virginia:

I agree with the authors that the failures are typical of stress corrosion cracking by residual static tensile stresses in the presence of environments specific for this type of failure. However, the authors, while they acknowledge the influence of caustic soda as a specific agent, express surprise at the rapidity of failure (less than 22 hours at 300 C). May I invite their attention to the article "Stress Corrosion Cracking of Austenitic Steels at Elevated Temperatures and Pressures" by V. P. Sidorov and A. V. Ryabchenkov of the Central Research Institute for Engineering Technology, U.S.S.R. This article is available as Translation No. 4250 from Henry Bratcher and was originally published in the June 1958 issue of *Metallovedenie i Obrabotka Metallov*.

The Russian authors tested three alloys, two of which bear a close resemblance

to AISI 321, in various caustic media with and without chloride contamination. At 330 C they report failure within 24 hours when the applied stress was above 45,000 psi. The "stress corrosion limit" (analogous to the endurance limit) is close to the yield point. Failures were predominately transcrystalline in nature. Some of the results are tabulated below:

Concentration	Time to Failure
30 Percent Caustic	2 hrs-25 min.
10 Percent Caustic	5 hrs-10 min.
5 Percent Caustic	6 hrs-30 min.
3 Percent Caustic	7 hrs-55 min.

The authors report failures at all caustic concentrations above 2 percent. No failures were encountered in straight sodium chloride solutions and the authors report that sodium chloride acts as an inhibitor in caustic under their test conditions.

We have encountered stress corrosion cracking of 347 equipment handling variable concentrations of caustic at elevated temperatures and pressures. It has not been possible to ascertain whether the failures were due to the caustic, to contaminants, or to prior service. On the other hand, our laboratory autoclaves have been used successfully in caustic work with only one thermowell failure (304) to date. A 48 hour laboratory test in 40 percent caustic at 225 C failed to cause cracking of Erichsen Cup impressions in annealed 304.

In our opinion, there is considerable room for further investigation of the phenomena of stress corrosion cracking of austenitic stainless steels in caustic services. Apparently, chloride contaminants alone are by no means the sole agents and may be no factor at all in some instances.

Reply by J. P. Hugo and L. G. Nel:

The authors wish to thank Mr. Dillon for his interesting comments and the information abstracted from Sidorov and Ryabchenkov's paper. We have not as yet had the opportunity to study this Russian paper, but it will doubtless be interesting to learn whether cracking was obtained under total or partial immersion conditions and whether cracking occurred in the vapor phase.

The question of whether or not concentration of the contaminant or corrodant is prerequisite for the occurrence of cracking is an important problem, since it perforce involves implications of a practical nature. We are, in general, in agreement with the opinion that there is considerable need for further investigation of the phenomena of stress corrosion cracking of austenitic stainless steels in caustic services.

Observations on the Mechanisms and Kinetics of Aqueous Aluminum Corrosion by R. L. Dillon. *Corrosion*, Vol. 15, No. 1, 13t-16t, (1959) Jan.

Comments by George Panter, Allis-Chalmers Manufacturing Company, Milwaukee, Wisconsin:

Mr. Dillon has emphasized that the bulk film is the rate determining factor in aqueous corrosion of aluminum in autoclaves. He also points out that in dynamic test experiments it was observed that as deionized water is equilibrated with aluminum oxide the water becomes less corrosive to aluminum. As a result of his investigations the following questions come to mind:

1. What influence will water equilibrated with aluminum oxide have on the nature of the bulk film such that corrosion is reduced?

2. What equilibrium of bulk film thickness can be predicted in a dynamic test which tends to remove the bulk film and yet is expected to reduce aluminum corrosion by using deionized water equilibrated with aluminum oxide?

Replies by R. L. Dillon:

1. Oxides formed in flowing refreshed water are more porous than those found on autoclave samples. A comparison of oxide porosity for X-8001 samples exposed simultaneously in the dynamic and low flow sections of a loop illustrates the point. The loop operated at 250 C with deionized water refreshed at the rate of 2 gph, the flow rate was 25 fps in the dynamic section, sample area: 96 sq inch dynamic section, 24 sq inch low flow section. Penetration rates are given in Table 1.

Both direct measurement of surface area per sample and high corrosion rates for dynamic samples with thick oxides point to the same conclusion. In the presence of flowing water, soluble constituents are leached out of the oxide rendering the oxide porous and non-protective.

Tests to determine corrosion rates at reduced or zero refreshment for dynamic systems are in progress.

2. In dynamic tests under conditions like those described in question 1, no equilibrium film is formed. Major losses of oxide occur not by dissolution, but by undercutting and detachment of the corrosion product in flakes. This can lead to erratic changes in average oxide thickness, even to reduction in apparent thickness with time. These changes in bulk oxide thickness are not reflected in changes in corrosion rate since the protective inner oxide is not involved.

TABLE 1—Dynamic and Low Flow Tests

Test Condition	Time	Surface Area, m ² /cm ²	Oxide wt/2 sq. in.	Penetration in Mills
Dynamic.....	1 Month	1.10	.1872	1.98
	2 Month	0.88	.2368	2.48
Low flow.....	1 Month	0.074	0.0960	0.50
	2 Month	0.065	0.1191	0.70

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Some interesting tests have been conducted recently at the Hanford Laboratories and independently at Chalk River which indicate that corrosion rates and aluminum corrosion product thickness can be substantially reduced by pre-autoclaving the samples prior to dynamic tests. It is likely that an aging process occurs which renders the corrosion product less subject to leaching and pore formation.

Influence of Service Temperature on the Resistance of Wrought Aluminum-Magnesium Alloys to Corrosion. *Corrosion*, Vol. 15, No. 2, 55t-62t, (1959) Feb.

Comments by S. K. Nowak, Aluminum Laboratories Limited, Kingston, Ontario, Canada:

The experimental results from the long time exposure programme are most welcome. Our results obtained during up to three years exposure agree entirely with those published by Dix et al.

The subject I would like to comment upon is the interpretation of continuous β phase precipitation in terms of nucleation theory. According to the authors, the formation of coarse precipitates is favored by both high aging temperatures (above 390 F-200 C) and low degrees of supersaturation. Our results indicate that the factor determining the morphology of precipitation is the dislocation-magnesium atom interaction. The following general mechanism of β phase precipitation in superpurity Al-Mg alloys can be proposed in terms of this interaction. In fully annealed alloys where the only regions of high dislocation density are grain boundaries, precipitation of the β phase occurs exclusively in those regions. In cold worked materials, however, two different processes control the precipitation simultaneously: the rate of recovery of the deformed matrix determined by the initial amount of cold work, and the degree of magnesium supersaturation.

In the case of low thermal energy, that is at 212 F, the nuclei appear first in the regions of high density of dislocations: grain boundaries, slip planes, deformation bands, etc. (Figure 1). Since at this temperature there is practically no annihilation and very little re-distribution of dislocations, the amount of precipitated phase increases with time, but its mode of distribution remains the same, (Figure 2). In the case of higher thermal energy, that is at 350 F (175 C) it seems that considerable re-distribution of dislocations and formation of subgrains occurs before the beginning of precipitation. For example, after 168 hours heat treatment, the material should be only mildly susceptible to stress corrosion (Figure 3). As time progresses, further re-distribution and annihilation of dislocations results in coalesced precipitate (Figure 4). By comparing Figure 2 with Figure 5 and Figure 4 with Figure 6, it can be concluded that the degree of supersaturation does not influence the mode of precipitation.

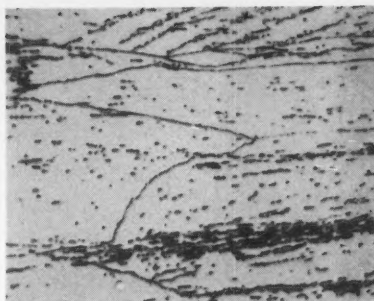


Figure 1—Al-4.7 percent Mg, 82 percent cold rolled, 168 hours at 100 C. 1320X.

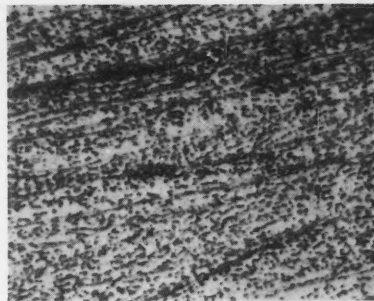


Figure 2—Al-4.7 percent Mg, 82 percent cold rolled, 7200 hours at 100 C. 1320X.

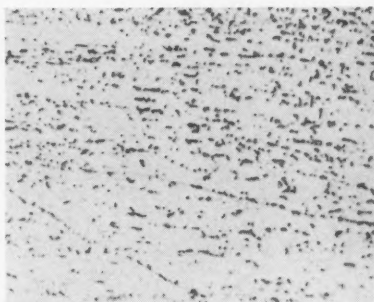


Figure 3—Al-4.7 percent Mg, 82 percent cold rolled, 168 hours at 175 C. 1320X.

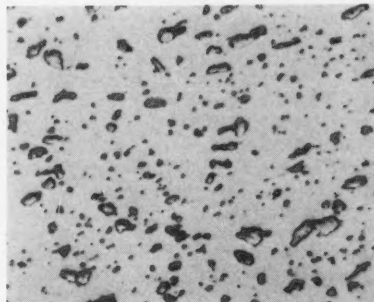


Figure 4—Al-4.7 percent Mg, 82 percent cold rolled, 7200 hours at 175 C. 1320X.



Figure 5—Al-9 percent Mg, 60 percent cold rolled, 8000 hours at 100 C. 1320X.

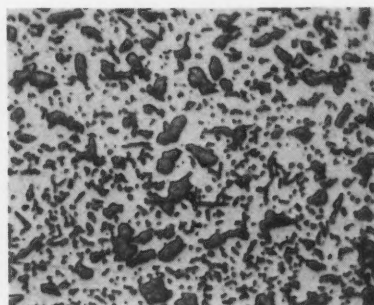


Figure 6—Al-9 percent, cold rolled, 8000 hours at 175 C. 1320X.

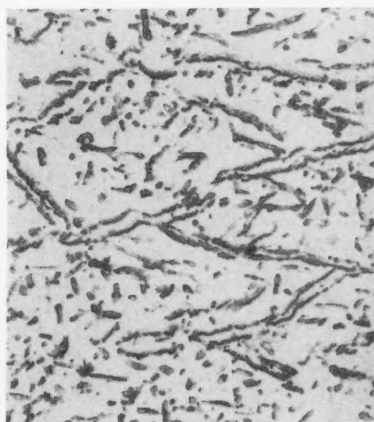


Figure 7—Al-9 percent Mg, 60 percent cold rolled, 8000 hours at 100 C. 4800X.

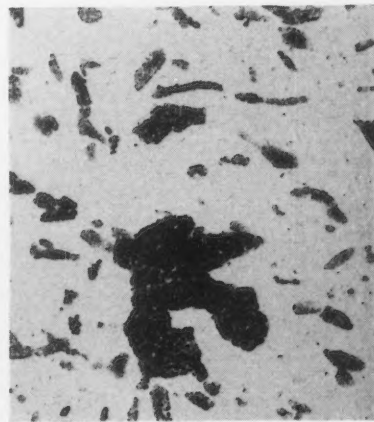


Figure 8—Al-9 percent cold rolled, 8000 hours at 175 C. 4800X.

Figures 7 and 8 clearly show the existence of two types of β phase particles: (1) At 212 F (Figure 7) the particles present are rod-shaped and oriented, which indicates at least partial coherence with the matrix. This precipitate probably represents the transition structure β' which has been shown to be very susceptible to stress corrosion; (2) At 350 F (Figure 8) the particles are coarse, no longer oriented and, therefore, not coherent with the matrix, which characterizes the final equilibrium structure of the β phase.

In the case of intermediate temperatures, that is 300 F (150 C), the material probably is extremely susceptible to stress corrosion after a heat treatment of a period of 168 hours. But after one year, when a re-distribution of dislocations has taken place and corrosion-sensitive β' has been transformed to the stable β phase, the resistance to stress corrosion improves.

It seems, therefore, that between 212 F and 350 F, the material must first pass through a dangerous susceptible condition before finally improving. Further work indicates that at 437 F (225 C), re-distribution and annealing of dislocations occur so quickly that irrespective

of the degree of magnesium supersaturation, no susceptible condition has time to occur. Incidentally, Figure 7 in the discussed paper provides confirmation of the above statements.

Reply by E. H. Dix, Jr., W. A. Anderson and M. Byron Shumaker:

We are indeed gratified to find from Dr. Nowak's discussion that the work done at the Aluminium Laboratories Limited at Kingston has substantiated our findings.

Dr. Nowak's interesting discussion on the precipitation mechanism was also appreciated since there is yet much to be learned concerning the various phases present and their effect on the resistance to stress corrosion of the Al-Mg alloys. Our work within the Al-Mg family of alloys has revealed susceptibility to stress corrosion by aging at 75 F, 200 F, 250 F, 300 F and 350 F. Perryman and Hadden*, working with higher Al-Mg alloys (Al-7 percent Mg), induced susceptibility to stress-corrosion cracking by aging treatments in the range of 435 F to 510 F. At all these temperatures the β' phase and the equilibrium β phase

*E. C. W. Perryman and S. E. Hadden, Stress Corrosion of Aluminum-7 Percent Magnesium Alloy. *J. Inst. Metals*, 77, 207, 1950.

apparently were involved either singly or in various combinations.

In our view, the susceptibility to stress corrosion in the Al-Mg alloys is not because a specific phase is precipitated such as the suggested coherent β' phase being precipitated at 212 F or that non-susceptibility occurs at 350 F or above because of the non-coherent equilibrium β phase being precipitated. Regardless of the phase present, the precipitate is believed to be highly anodic. When we integrate all of our data we find that regardless of the magnesium level in the alloy if aging or thermal treatments can produce a microstructure where the precipitate is substantially continuous along the grain boundaries (without considerable precipitate within the grain bodies) the alloy is highly susceptible to stress-corrosion cracking.

It is felt that the improvement in resistance to stress corrosion after prolonged heating at the intermediate temperatures, such as 300 F, is the result of general precipitation within the grain bodies increasing the ratio of the area of the anodic phase to that of the cathodic solid solution. The attack is then spread out over a larger area rather than being concentrated at the grain boundaries.

Discussions to technical articles appear in the June and December issues only when they do not immediately follow the article to which they pertain. Discussions received from October through March will appear in the June issue and those from April through September in the December issue.

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TECHNICAL COMMITTEE ACTIVITIES

210,000 Copies of Technical Reports Distributed in 1958

Over 210,000 copies of 12 technical committee reports were distributed in 1958, according to the report of the Technical Practices Committee submitted to the NACE president.

A total of 36 committee meetings were held since the 1957 report of this committee.

Technical committee membership increased 25 during the past year. Total membership in 1957 was 1312; in 1958, 1337.

New Activities Begun

Thirteen new activities were begun in 1958: three new units and 10 new task groups. New units formed were T-2L, Wax-Type Pipe Coatings and Component Wrappers, T-9A Biological, and T-9B Preservatives and Their Appraisal.

New task groups formed were T-1K-3 Inhibitor Testing at Elevated Temperatures, T-2H-5 To Review and Encourage Publication of Technical Articles on Asphalt-Type Protective Coatings, T-4A-3 Methods and Materials for Grounding, T-6A-19 Asphalts, T-6A-20 Plastisols, T-6B-15 Silicones, T-6D-4 Specifications for Shop Cleaning and Priming, T-6D-5 Exploratory Task Force on Painter Safety and Painter Education, T-8A Chemical Cleaning and T-8B Refinery Industry Corrosion Los Angeles Area.

Committees Dissolved

The following task groups were dissolved during 1958: T-1C-3, T-1C-4, T-1D-1, T-1H-1, T-1H-3, T-1H-4, T-2H-1, T-3F-1, T-3F-4, T-3F-5, T-4A-1, T-4A-2, T-5B-3, and T-5B-5. Unit Committee T-1J on Oil Field Structural Plastics was dissolved also because the committee considered the unit's purpose had been fulfilled.

Co-operation With Other Organization

Twelve areas of co-operation with other organizations were reported during 1958: ASTM Joint Committee on Chemical Analysis by Powder Diffraction, API Panel on Cooling Water, ASTM Committee D-19 on Industrial Water, Society of the Plastics Industry, ASTM Committee A-10 on Methods of Corrosion Testing, National Association of Fan Manufacturers, Steel Structures Painting Council, Corrosion Committee of the Federation of Paint and Varnish Production Clubs, Construction Specifications Institute, American Welding Society's Committee on Metallizing, API Division of Refining Subcommittee on Corrosion, and the Inter-Society Corrosion Committee.

Four New Coordinating Committees

Four coordinating committees affiliated with three NACE units during 1958. Northeast Region Corrosion Coordinating Committee T-7A was joined by the Columbus and Central Ohio Committee on Corrosion.

South Central Region Corrosion Coordinating Committee T-7D was joined by the San Antonio Area Corrosion Committee.

Western Region Corrosion Coordinating Committee T-7E was joined by the Central California Catholic Protection Committee and San Francisco Committee on Corrosion.

Water Production's Effect on Corrosion Discussed by T-1D

Unit Committee T-1D, Sour Oil Well Corrosion, discussed activities of its Task Group T-1D-2, West Texas-New Mexico, methods of checking corrosion and effect of water production on corrosion.

Task Group T-1D-2

Activities of Task Group T-1D-2 have centered around a study covering inhibitors, pump corrosion, sucker rods and tubing, plastic pipe and tanks, storage tanks and treaters. Information was obtained by a questionnaire sent to 100 operators with 20 returned. These covered 8992 wells of which 2971 were considered corrosive.

Of the wells being tested with inhibitors, 744 were treated with oil soluble inhibitors, 466 with oil soluble water dispersible and 148 with water soluble. Inhibitor ratio to total fluid varied from 1 quart for each 10 barrels of fluid to 1 quart to 800 barrels of fluid. Inhibitor ratio to oil varied from 1 quart to 10 barrels of oil to 1 quart to 70 barrels of oil.

Treating frequency varied from daily to once each 30 days. The decision to start treatment was most often determined by well service records although some operators used coupon studies and offset operators' practices.

Storage tanks, based on a weighted average of the number of wells being reported on, were 70 percent welded tanks, 14 percent bolted galvanized steel, 11 percent wooden tanks and 5 percent bolted black steel. By the same averages, coatings for tanks were 46 percent coal-tar epoxy, 31 percent vinyl, 16 percent epoxy and 7 percent coal tar.

The survey showed that heater treaters were used for 64.3 percent of the treating equipment, wooden gun barrels for 19 percent and steel gun barrels for 16.7 percent.

Cathodic protection was used on 62.5 percent of the heater treater, coatings on 2.4 percent and no protection for 35.1 percent.

Coatings were used to protect 58.7 percent of the steel gun barrels, cathodic protection on 37 percent and no protection on 4 percent.



Cathcart



Saffian

T-6F CHAIRMEN recently elected are Chairman W. P. Cathcart of Oak Ridge National Laboratories, Oak Ridge, Tenn., Vice Chairman B. M. Saffian of General Dynamics Corp., Groton, Conn., and Secretary Paul W. Lewis of the Bureau of Reclamation, Denver Federal Center, Denver, Colo. This committee investigates applications and methods of protective interior linings.

Methods of Checking Corrosion

Coupons and service records were shown by the West-Texas-New Mexico questionnaire to be the main criteria of checking on corrosion. In its discussion, T-1D members stated that bottom hole coupons were more valuable than surface coupons, but field personnel disliked bottom hole coupons because of the difficulty in running them.

The main use of coupons was suggested to be in comparative values. Comparisons can be used before and after inhibitor treatment or to indicate a comparison between different inhibitor treatments or to indicate changes in corrosive characteristics of the well during its productive life.

Effect of Water Production on Corrosion

Effect of water production on corrosion was discussed briefly. Cases were mentioned in which corrosion almost disappeared at high water production rates. In some West Texas wells producing little water had caused severe

(Continued on Page 74)



Kunkel



Copson

T-5E CHAIRMEN recently elected are Chairman E. V. Kunkel of Celanese Corporation of America, Bishop, Texas, and Vice Chairman Harry R. Copson of International Nickel Co., Inc., Bayonne, N. J. This committee deals with stress corrosion cracking of austenitic stainless steels.

Inhibitor Squeeze Treatment, Coating Reports Heard by T-1C

Technical Unit Committee T-1C, Sweet Oil Well Corrosion, heard reports at an October meeting in New Orleans from its task group on inhibitor squeeze treatment, plastic coatings and plastic coatings for paraffin prevention.

Inhibitor Squeeze Treatment

Inhibitor squeeze treatment was classified as short, medium and long squeeze. The short squeeze consisted of pumping chemical or chemical mixture to the tubing string bottom without forcing intentionally the fluid into the formation. Medium squeeze consisted of forcing chemicals into the formation by over-flushing it with 1 to 50 barrels of fluid. The long squeeze required an over-flush of at least 51 barrels of fluid.

Over 300 wells were given a total of 520 squeeze treatments. Of these, 24 were considered failures in regard to corrosion control. The remaining 276 wells (or about 92 percent) were considered successful. Success or failure was based on iron counts and/or caliper surveys.

No definite procedure is available to determine which squeeze treatment should be given to a well.

Detrimental production changes were noted in 8 wells which received squeeze treatment. Five of these were caused by water sensitive sands. Production was remedied by an immediate oil squeeze or the wells were allowed to return to

normal production in 30 to 60 days. One production change was caused by use of improper chemicals; another caused by cleaning action on perforation.

Generally, well production was affected for about four or five days. Several wells were reported to have been stimulated by the cleaning action of the chemicals. Most of the wells, however, were unaffected by squeeze treatment.

Pump equipment costs varied from \$30 to \$960 per well. Pumping costs, without inhibitor costs) can be correlated with the well-head pressures:

0 to 200 psi: \$30 to \$80 per well depending on depth pumping rate, amount of over-flush, pumping rates, etc.

200 to 300 psi: \$50 to \$150 per well depending on above factors.

3000 to 8500 psi: \$150 to \$300 per well. Normally, one drum of regular inhibitor proved to be the minimum amount of inhibitor per squeeze. Two to ten drums were used in several wells.

Plastic Coating

A member reported on plastic coating of various types of tubing joints such as 8-round, Spang-Seal, X-Lime, Gray-Lock, Hydril CS and Hardy-Griffin.

Coatings for Paraffin Prevention

A member stated that a plastic coating, a 5-coat baked-on phenolic, was used for paraffin prevention. It was tested in a sweet, non-corrosive oil field of 133 wells of which 90 were natural flowing, 8 gas-lift and 17 pumping.

The coating proved effective in all types of wells, according to reduction of scraping costs. Effectiveness varied from 80 percent in natural flowing wells and 94 percent in pumping wells to 100 percent in gas-lift wells.

Another test to evaluate economics of paraffin prevention methods indicated that plastic coating is the most economical. The other methods included coating, paraffin solvents, mechanical scrapers, hot chemicals and hot oil.

T-1K Hears Reports From 3 Task Groups

Technical Unit Committee T-1K, Inhibitors for Oil and Gas Wells, heard reports from its task groups at a meeting held last October in New Orleans.

T-1K-1

Task Group T-1K-1 on Sour Crude Inhibitor Evaluation reported that a revised static test to screen sour corrosion inhibitors had been proposed and approved to replace the earlier test. The sour inhibitor dynamic test was reviewed, also.

T-1K-2

Task Group T-1K-2 on Sweet Crude Inhibitor Evaluation reported that agreement had been expressed at the Houston meeting that any good dynamic sweet corrosion inhibitor test can be used for evaluating sweet inhibitors.

Future work of the task group will be in consideration of film persistence.

T-1K-3

Task Group T-1K-3 on Inhibitor Test-

ing at Elevated Temperatures presented a proposal by the Texas Research Associates to conduct research on the high temperature corrosion of steel in systems encountered in producing and processing oil and gas. The NACE committee is being canvassed to determine the amount of interest in supporting this proposal by company contributions.

Approval Given for New Unit Under T-1

The Technical Practices Committee approved, at the Chicago Conference, formation of a new unit committee under Group Committee T-1, Corrosion of Oil and Gas Well Equipment. The new committee will be designated T-1E, Corrosion in Water Injection Systems.

Cecil O. Smith of Carter Oil Co., Tulsa, Okla., and Joy T. Payton of the Texas Company, Houston, Texas, have been elected chairman and vice chairman respectively.



Smith



Payton

The committee's work will deal specifically with corrosion of equipment in waterflood and salt water disposal systems, means of mitigation of such corrosion and investigation into the characteristics of the various oil field brines and source water used for waterflood purposes which contribute to corrosion.

Three task groups have been appointed for the committee with the following chairmen:

Task Group 1: Classification of Corrosive Waters. Chairman L. C. Case, Gulf Oil Corp., Tulsa, Okla.

Task Group 2: Inhibitor Treatment of Injection Water. Chairman John R. Bruce, Shell Oil Co., Tulsa, Okla.

Task Group 3: Corrosion Resistant Materials for Water Injection Systems. Chairman R. S. Ladley, Phillips Petroleum Co., Bartlesville, Okla.

This new committee held two organizational meetings at New Orleans and Tulsa last year. Its meeting in Chicago was its first meeting at which technical reports from task groups were heard.

Water Production—

(Continued From Page 73)

trouble, possibly from drawing air into the pump.

Records from one field that a peak corrosion rate was obtained between 5 and 15 percent water. Between 15 and 45 percent water, the corrosion rate dropped off, and at about 50 percent water it started upward again. A protective scale seemingly was formed at certain stages, and when the scale stopped forming, the corrosion rate went up again.

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NACE NEWS

1960 Corrosion Show Brochures Are Mailed

Descriptive brochures concerning the 1960 Corrosion Show to be held at Dallas March 15-17 were mailed May 1 to firms on the prospect list for the NACE shows. Several contracts for space have been received already.

R. W. Huff, Jr., NACE exhibits manager said, because of the limited amount of exhibit space available at the Dallas auditorium, he expected the show to be sold out before fall. "Companies that are interested in exhibiting at the 1960 Show should send in contracts at once," he said. Mr. Huff also said he has a supply of exhibit brochures and contracts ready to mail to anyone interested who did not receive a copy in the May mailing.

MIT Short Course Final Plans Made For June 22-26

Lecturers and discussion topics have been scheduled for the one-week Special Summer Program in Corrosion to be held June 22-26 at Massachusetts Institute of Technology, Cambridge.

Five course subjects and their lecturers are listed below:

1. Electrochemical Theory: H. H. Uhlig, professor of metallurgy and director of MIT Corrosion Laboratory.
2. Interpretation and Application of Polarization Measurements: Milton Stern, Metals Research Laboratories, Niagara Falls, N. Y.
3. Surface Chemistry, Inhibitors and Passivators: Norman Hackerman, head of chemistry department at the University of Texas, Austin.
4. Metallurgical Factors: Bruce Chalmers, professor of metallurgy in Division of Engineering and Applied Physics, Harvard University, Cambridge.
- 5a. Fundamentals of Oxidation and Tarnish: H. H. Uhlig.
- 5b. Corrosion Resistant and Oxidation Resistant Metals and Alloys: Milton Stern and H. H. Uhlig.

The program is designed for those who have had previous instruction in corrosion such as various short courses sponsored by NACE or who have been in corrosion work and who wish to enlarge their understanding of fundamentals. Students should have previous acquaintance with elementary physical chemistry.

Tuition is \$200 payable upon notification of admission. No academic credit is offered.

H. H. Uhlig is in charge of the special program.

3 Exhibits on Corrosion Win Prizes in School Science Fairs

Three exhibits pertaining to corrosion have won prizes in high school science fairs in Washington, D. C., California and Texas. Prepared by high school students, each exhibit received cooperation from NACE members.

The three exhibit winners are Susan Weiser of Washington, D. C., Judy Minna of San Diego, Cal., and Judy Seastrand of Houston, Texas.

Susan Weiser won first prize in chemistry, grand prize for all eighth graders in the area and second place in the Taft Junior High School Science Fair. Her exhibit dealt with cathodic protection. She was put in contact with John Loeffler, chairman of the South Central Region's Educational Committee by her letter to Tennessee Gas Transmission Company of Houston. Mr. Loeffler supplied information, addresses of resources and other materials for the exhibit.

Mr. Loeffler also gave resource information to Judy Seastrand of Houston for her exhibit on plastics. She won a ribbon and trophy for her first place in the miscellaneous division of the Science Fair held at Cullen Junior High in Houston. She made part of the exhibit showing the uses of liquid vinyl poured into molds and baked at 350 F for 30 to 45 minutes.

Judy Minna won first place in the girls chemistry division at the science fair in San Diego, Cal. A 16-year old junior at Rosary High School, Judy was one of 200 participants. Technical advice and reference material for her exhibit on corrosion were furnished by F. O. Waters, corrosion engineer for the San Diego Utilities Department and past chairman of the NACE San Diego Section.



FIRST CORROSION PROJECT at a high school science fair at San Diego, Cal., won first place for Judy Minna in the girls chemistry division. A 16-year old junior at Rosary High School, Judy was one of 200 participants. Technical advice and reference material for her exhibit were furnished by F. O. Waters, corrosion engineer for the San Diego Utilities Department and past chairman of the NACE San Diego Section. The Science Fair was held in Balboa Park April 3-7.

Corrosion's Front Cover Is Changed for First Time

Format for the front cover of CORROSION has been changed effective with this (June, 1959) issue. This represents the first major change in format since the magazine was established in 1945, earlier modifications having been limited in scope.

The principal difference is that the former practice of using a photographic illustration has been abandoned. The characteristic green background has been retained in the form of a strip down the left side and a selected list of titles of articles in the issue has been added. The word "Corrosion" which has appeared on all issues is retained unchanged.

This change was authorized at a May 4 meeting of the Publication Committee at Houston in the belief that the new format will be more useful to readers. It was thought that readers will find the selected items from the contents

(Continued on Page 77)

'61 NACE Conference Chairman Appointed

Walter A. Szymanski of Hooker Chemical Corporation has been appointed general chairman of the 1961 Annual NACE Conference scheduled for March 13-17 at the Hotel Statler in Buffalo, N. Y.

Mr. Szymanski is head of the Corrosion and Materials Testing Laboratory in the Central Engineering Division of his corporation. A native of

Buffalo, he attended the Erie County Technical Institute and University of Buffalo. In 1958 he was chairman of the Niagara Frontier Section and is a member of several NACE technical committees. He has written several technical papers on corrosion.



Szymanski

News intended for publication in CORROSION should be in Houston not later than the 10th of the month preceding month of publication.

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No. 910 Gray, 12 mils.	13.66	12.20	11.02	9.76	8.54	8.04	7.62
No. 940 Black, 12 mils.							
No. 940 Gray, 12 mils.							
No. 920 Black, 20 mils.	19.30	17.22	15.56	13.78	12.06	11.38	10.76

PIPELINE ROLL—3 inch I.D. Core—400, 600, 800 ft. length—Price Per Square, Dollars

Quantity, Squares	Less than 102	102 to 500	501 to 1499	1500 to 2999	3000 to 5000	5001 to 25001	25002 and Over
No. 900 Black, 14 mils.	13.66	12.20	11.02	9.76	7.90	7.44	7.04
No. 920 Black, 20 mils.	19.30	17.22	15.56	13.78	11.16	10.52	9.95



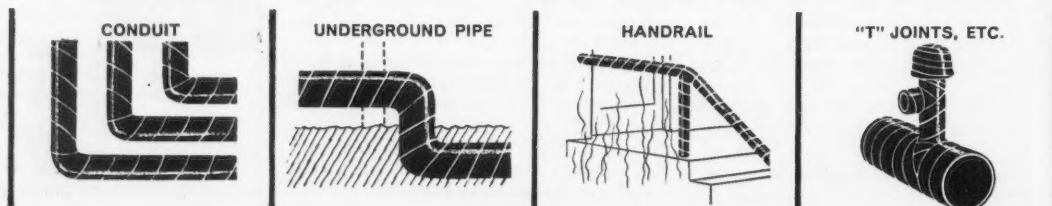
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Western Region News



PORTLAND SECTION held a Pulp and Paper Symposium at its April meeting. Shown above are John Van Bladeren, Northwest Natural Gas Co., Portland, chairman; Ed M. Read, Longview Fibre Co., Longview, Wash., guest speaker; Dean D. Burgan, Electric Steel Foundry, Portland, moderator; W. R. Shimmin, Crown Zellerbach, Camas, Wash., guest speaker; Otto Hudrik, Flox Co., Portland, vice chairman; and George H. Beisse, Weyerhaeuser Pulp Division, Longview, Wash., guest speaker.

Portland Section heard Melvin Romanoff, National Bureau of Standards, speak on soil corrosion at the May 14 meeting.

The section's June meeting will be a round-robin at which each commercial representative who is a section member will be permitted to tell about his company and its products.

San Francisco Bay Area Section will have its summer recess during June, July and August. No meetings are scheduled.

Western Region Conference will be held September 29-October 1 at the Bakersfield Inn, Bakersfield, Cal.



SECTION CALENDAR

June

- 3 Teche Section. Short Course Banquet at the Petroleum Club.
- 5 Birmingham Section.
- 8 Central Oklahoma Section. Ladies Night. Tropical Cafeteria, Oklahoma City.
- 18 Teche Section. Regular meeting at Petroleum Club.

July

- 30 Teche Section. Petroleum Club.

Corrosion's Cover—

(Continued From Page 75)

helpful in that they can quickly tell whether or not the issue contains articles of immediate interest.

Other decisions also were made concerning CORROSION and other NACE publications at the meeting.

Lapel pins approximately 7/16 inches high, made of gold with inlaid red enamel and a ruby center, are available to NACE members only. Price is \$10. Address orders to Executive Secretary, 1061 M & M Bldg., Houston 2, Texas.

Canadian Region News

Edmonton Section held its April 23 meeting on the subject of pipeline coating inspections. Guest speaker was Gordon Russell of the Interprovincial Corrosion Control Company.

The section's annual mixed social was held May 7 with guests invited.

Edmonton Section educational activities for the year have included co-operation with the ASM in contributing the corrosion section at the ASM course on metallurgy and related topics.

Transactions of the 1957 Corrosion Conference, an educational program held in conjunction with the 1957 Canadian Region Conference, are now available in printed form. The theme was the Six Keys to Corrosion Control; these keys were structural design, protective coatings, environmental treatment, inhibitors, corrosion resistant materials and cathodic protection. Copies are available from J. Gattenmeyer, Canadian Protective Coating Ltd., 9420 91st St., Edmonton, Alberta, Canada, at \$2 per copy.

North Central Region News

Kansas City Section held a regular meeting on May 11 with the subject for discussion on corrosion problems of underground structures in urban and metropolitan areas. Speakers were A. C. Singer, Gas Service Co., C. E. Gilliam, Kansas City Power & Light, and Forrest E. Davidson, Southwestern Bell Telephone Co.

Special Meeting of the North Central Region will be held July 1 in Cleveland. Guest speaker will be T. P. Hoar, professor of metallurgy at the University of Cambridge, Cambridge, England. Dr. Hoar received the 1957 Whitney Award.

The 1959 North Central Region Conference will be held October 20-22 in Cleveland, Ohio.

Northeast Region News

Speakers Are Scheduled For Oct. 5-8 Conference

Speakers from universities and government research laboratories have been scheduled to participate in the October 5-8 Northeast Region Conference in Baltimore. Also, 22 speakers representing industrial laboratories will present papers during the conference.

Ten papers more than could be included in the program were received, according to Herman S. Preiser, chairman of the technical program.

Speakers on the program will represent Oak Ridge National Laboratory, Rutgers University, Bureau of Ships, Naval Research Laboratory, University of Pennsylvania, Los Alamos Laboratory, National Bureau of Standards, University of Illinois, Boston Naval Shipyard, U. S. Naval Engineering Experiment Station and the U. S. Coast Guard.

1959 Northeast Region Conference will be held October 5-8 at the Lord Baltimore Hotel, Baltimore, Maryland.

4th Annual Corrosion Control Short Course sponsored by the Miami Section will be held November 16-20 at the Key Biscayne Hotel, Miami, Florida.

The University of Illinois Corrosion Control Short Course will be held on the Urbana Campus December 7-11.

Niagara Frontier Section held a panel discussion on combating corrosion for its March 10 meeting, at the invitation of the Buffalo Forge Company. Topics included introduction to corrosion, vinyl and epoxy coatings, plastics, plastisols and baked coatings, zirconium, tantalum and alloys, rubber, stainless steel, aluminum and copper alloys and hi-nickel alloys.

Panel members included E. H. Caldwell, R. H. Shaffner, L. T. Kasmer, E. H. Robb, J. S. Gleason, E. L. Lockman and G. L. Cox.

Kenneth M. Huston has been appointed co-chairman of the Northeast Region Conference to be held in Baltimore, October 5-8. Mrs. Doris M. Lewis has been appointed chairman of the ladies committee. Her assistants will be Mrs. A. C. Burton and John A. Hendricks.

Plans for the ladies program include sight-seeing tours of Baltimore, Fort McHenry, Annapolis and the U. S. Naval Academy, coffee hours each morning and luncheons on the first two days of the conference. An oyster roast and banquet have been scheduled for the conference also.



Huston

1963 Annual Conference Changed to Atlantic City

The 19th Annual NACE Conference and 1963 Corrosion Show will be held March 18-22, 1963, at the Chalfonte-Haddon Hall Hotel in Atlantic City, N. J.

Preliminary arrangements have been made for easy air transportation to Atlantic City from all points.

The 1963 conference was originally scheduled to be held in Philadelphia.

NACE Certificates of Membership are available from the Central Office. Measuring $5\frac{1}{2}$ x $8\frac{1}{2}$ inches, the certificates cost \$2 each and are signed by the association president and executive secretary.

Certificates for past chairmen of regions and sections, measuring 9 x 12 inches, are available from the Central Office at \$7.50 each.

NEWS DEADLINE FOR CORROSION

News intended for publication in CORROSION should be in Houston not later than the 10th of the month preceding month of publication. When events occur at or near this date, it sometimes is possible to hold space for news stories, provided advance notice is given. An estimate of the amount of space required should be included in the advance notice.



NATIONAL and REGIONAL MEETINGS and SHORT COURSES

1959

Sept. 29-30, Oct. 1—Western Region Conference. Bakersfield Inn, Bakersfield, Cal.

Oct. 1-2—Southeast Region Conference. Jacksonville, Florida, Robert Meyer Hotel.

Oct. 5-8—Northeast Region Conference. Lord Baltimore Hotel, Baltimore, Md.

Oct. 12-15—South Central Region Conference. Denver, Colo. Cosmopolitan Hotel.

Oct. 20-22—North Central Region Conference. Cleveland, Statler Hilton Hotel.

1960

February—Canadian Region Western Division. Vancouver.

March 14-18—16th Annual Conference and 1960 Corrosion Show. Dallas, Texas, Memorial Auditorium.

Oct. 11-14—Northeast Region Conference. Huntington, W. Va.

Oct. 19-20—North Central Region Conference. Milwaukee.

Oct. 25-28—South Central Region Conference. Mayo Hotel, Tulsa.

1961

March 13-17—17th Annual Conference and 1961 Corrosion Show, Buffalo, N. Y., Hotel Statler.

Oct. 9-11—North Central Region Conference. St. Louis, Chase Park Plaza Hotel.

Oct. 24-27—South Central Region Conference. Houston, Shamrock Hotel.

Oct. 30-Nov. 2—Northeast Region Conference. New York City, Hotel Statler.

1962

March 18-22—18th Annual Conference and 1962 Corrosion Show. Kansas City, Municipal Auditorium.

October 16-19—South Central Region Conference. Hilton Hotel, San Antonio, Texas.

SHORT COURSES

1959

June 2-4—Teche Section. Corrosion Control Short Course, Southwestern Louisiana Institute, Lafayette.

June 2-4—Appalachian Underground Corrosion Short Course, West Virginia University, Morgantown.

June 22-26—Massachusetts Institute of Technology Advanced Short Course in Fundamentals of Corrosion Reactions and Corrosion Control, Cambridge.

November 16-20—4th Annual General Florida Conference 1959 Corrosion Short Course. Key Biscayne Hotel, Miami.

December 7-11—University of Illinois Corrosion Control Short Course. Urbana Campus.



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South Central Region News

Charles A. Geddes, Jr., 54, NACE member and corrosion engineer for Cathodic Protection Service of Houston, was killed March 21 in an accident on an offshore rig at Abbeville, La. He had been with the company 7 years.

Central Oklahoma Section will hold a special ladies night meeting on June 8 at the Tropical Cafeteria in Oklahoma City.

At the April 13 meeting **J. Wade Watkins** of the U. S. Bureau of Mines spoke on application of dissolved gas corrosion research to waterflood problems.

North Texas Section held a special ladies night program May 11 as its last meeting before summer adjournment. The film "Oil and Gas Well Corrosion" was shown.

Shreveport Section heard a panel discuss economics of cathodic protection of underground pipelines at the May 5 meeting. Panel members were **Pat Miller**, Texas Eastern Transmission Corp., **H. V. Beezley**, United Gas Pipeline Corp., **Frank Therrell**, Interstate Oil Pipeline Co., **N. J. Olive**, Arkansas Fuel Oil Corp., and **J. J. Wise**, Ark-La Gas Co.

Ned Stearns has been appointed chairman of the short course to be held in September. **Grady Howell** is chairman of the annual barbecue scheduled June 9. **John Graves** was appointed chairman of the annual ladies night scheduled for July.

Teche Section will hold its regular meeting on June 18 at the Petroleum Club, Lafayette. The short course banquet is scheduled for June 3.

E. J. Sablatura of Gulf Oil Corporation spoke on his company's experience with inhibitor squeeze at the April 29 meeting.

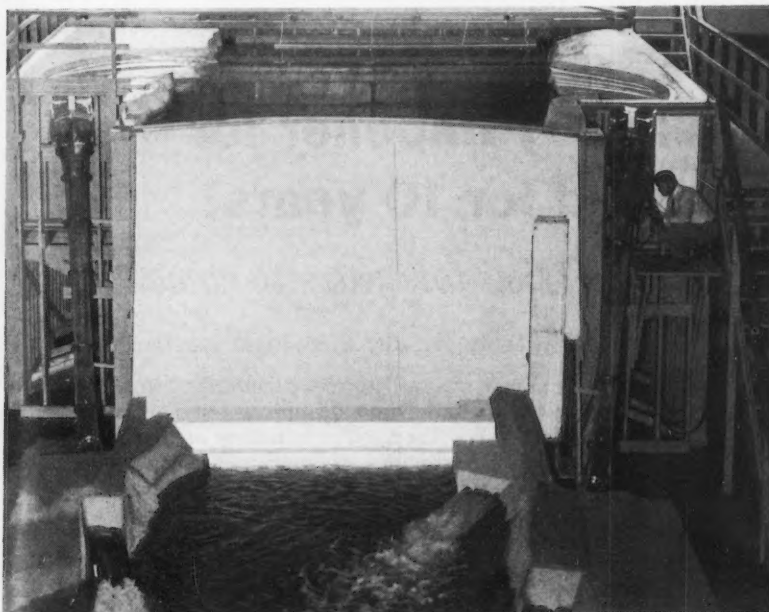
Corpus Christi Section heard **Gordon Doremus** of Cathodic Protection Service, Houston, speak on instruments for corrosion measurement at the April 28 meeting.

Permian Basin Section April 14 meeting was a discussion of inhibitor screening tests given by **George E. Purdy** of the Tretolite Company.

East Texas Section heard **Joe Rench**, vice president of Napko Corp., Houston, speak on plastic material for corrosion control at the April 28 meeting.

Houston Section heard a panel discuss testing, application and inspection of spray applied, chemical resistant lining materials at the April 14 meeting. Panel members were **Roland Foster**, Dow Chemical Co., **Freeport**, **Barney Goodman**, Tube-Kote Inc., Houston, **Parker Helms**, Union Carbide Chemicals Co., Texas City, **Lee Mangrum**, Linco, Inc., Houston, and **Frank Wilson**, Clemtex, Inc., Houston. **Mr. Wilson** was panel moderator.

South Central Region's 1959 Conference will be held October 12-15 at the Cosmopolitan Hotel in Denver, Colo.



HYDRAULIC MODEL of the Glen Canyon Dam to be seen during the 1959 South Central Region Conference in Denver, October 12-16, is shown above. This and other models will be shown during a tour of the Bureau of

Reclamation Engineering Laboratories. It is a full working scale model of the dam now under construction on the Colorado River above Hoover Dam. The dam will be 700 feet high, second only to Hoover Dam in this country.



TECHNICAL REPORTS

on

PROTECTIVE COATINGS

Other Than Pipe Line Coatings

T-6A Application Techniques, Physical Characteristics and Corrosion Resistance of Polyvinyl Chlor-Acetates. A Report of Unit Committee T-6A on Organic Coatings and Linings for Resistance to Chemical Corrosion. Publication 54-4. Per Copy \$5.00.

T-6A Report on Rigid Polyvinyl Chloride. A Report of NACE Technical Unit Committee T-6A on Organic Coatings and Linings for Resistance to Chemical Corrosion. Publication 56-4. Per Copy \$5.00.

T-6A Report on Epoxy Resins. A Report of NACE Technical Unit Committee T-6A on Organic Coatings and Linings for Resistance to Chemical Corrosion. Publication 56-5. Per Copy \$5.00.

T-6A Report on Application Techniques, Physical Properties and Chemical Resistance of Chlorinated Rubber Coatings. NACE Technical Unit Committee T-6A on Organic Coatings and Linings for Resistance to Chemical Corrosion. (Pub. 56-6.) Per Copy, \$5.00.

T-6A Vinylidene Chloride Resins. A Report of Unit Committee T-6A Prepared by Task Group T-6A-3 on Vinylidene Chloride Polymers. Publication No. 57-9. Per Copy \$5.00.

T-6A Reinforced (Faced) Tank Linings. A Report by NACE Committee T-6A. Pub. 58-7. Per Copy \$5.00.

T-6A Report on Thermoplastic Cool Tar Base Linings. A Report of NACE Unit Committee T-6A on Organic Coatings and Linings for Resistance to Chemical Corrosion, prepared by Task Group T-6A-16 on Coal Tar Coatings. Pub. 58-10. Per Copy \$5.00.

T-6B Protective Coatings for Atmospheric Use: Their Surface Preparation and Application Physical Characteristics and Resistance. A Report of Unit Committee T-6B on Protective Coatings for Resistance to Atmospheric Corrosion. Publication No. 57-10. Per Copy \$5.00.

TP-6G First Interim Report on Recommended Practices for Surface Preparation of Steel. (Pub. 50-5.) Per Copy, \$5.00.

TP-6G Second Interim Report on Surface Preparation of Steels for Organic and Other Coatings. (Pub. 53-1.) Per Copy, \$1; five or more copies to one address, per copy \$5.00.

T-6K Acid Proof-Vessel Construction with Membrane and Brick Linings. A Report of NACE Technical Unit Committee T-6K on Corrosion Resistant Construction with Masonry and Allied Materials. Publication No. 57-6. Per copy \$5.00.

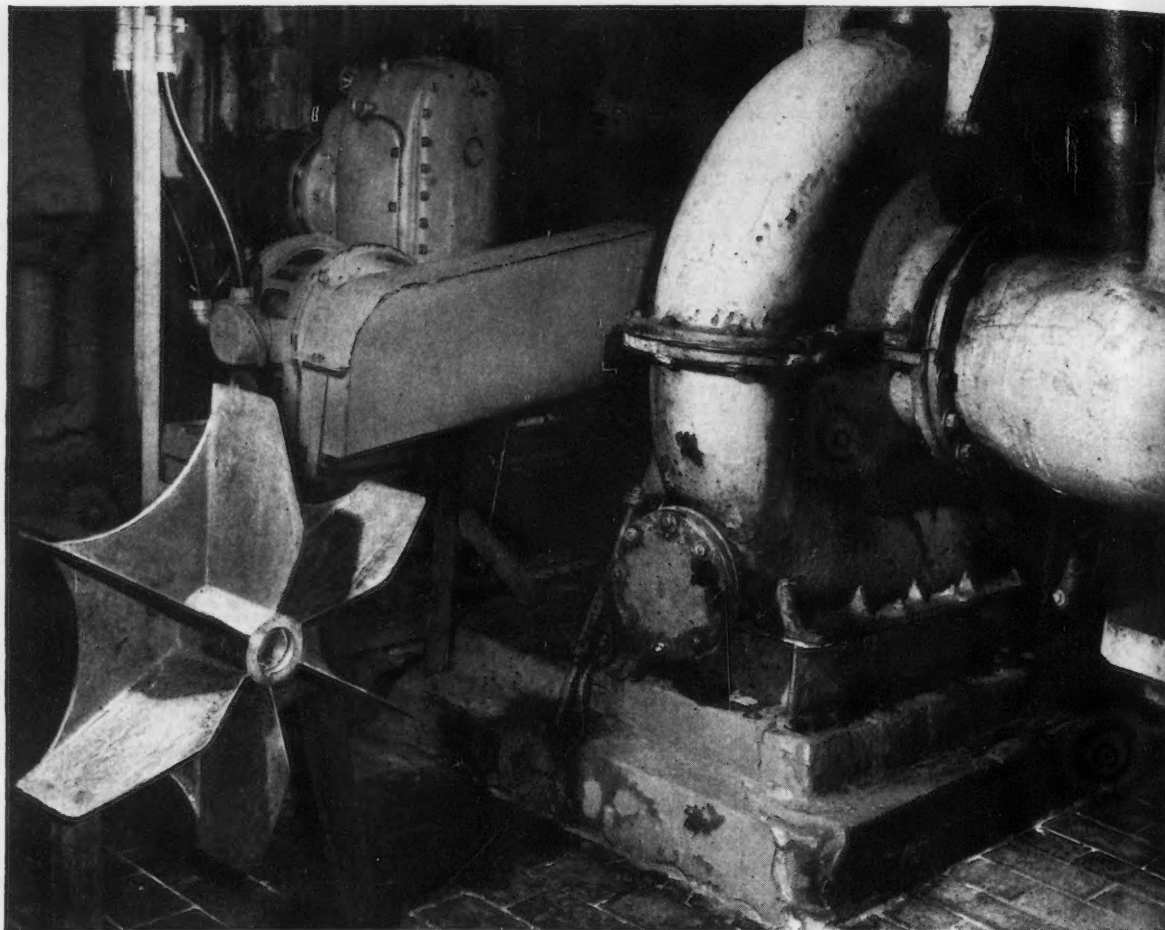
Remittances must accompany all orders for literature the aggregate cost of which is less than \$5. Orders of value greater than \$5 will be invoiced if requested. Send orders to National Association of Corrosion Engineers, 1061 M & M Bldg., Houston, Texas. Add 65¢ per package to the prices given above for Book Post Registry to all addresses outside the United States, Canada and Mexico.

NATIONAL ASSOCIATION OF CORROSION ENGINEERS

1061 M & M Bldg. Houston 2, Texas

NACE's 16th Annual Conference and 1960 Corrosion Show will be held March 14-18 in the Memorial Auditorium, Dallas, Texas.

4th Annual Corrosion Control Short Course sponsored by the Miami Section will be held November 16-20 at the Key Biscayne Hotel, Miami, Florida.



The impeller you see has been a spare for 10 years! Both it and the working impeller inside the housing at right are Illium "G", a 56% nickel alloy par-

ticularly resistant to the SO_2 gases handled by the blower. The blower is installed at the Crown-Zellerbach Corporation plant in Camas, Washington.

Spare nickel alloy impeller for SO_2 sits unused for 10 years!

... working impeller's still good for years to come


SO_2 gases are mighty hard on most impeller metals. Yet, the spare impeller you see has not been needed since the blower went in over 10 years ago! Blower exhausts 17% SO_2 gases at 90°F from pulp mill sulfur burners.

Both impellers are Illium "G"*, a high-nickel alloy cast by Stainless Foundry and Engineering, Inc., Milwaukee, Wisconsin. This high-nickel alloy provides superior resistance to the corrosive attack of many strong acids (particularly sulfur compounds), caustics, oxidizing and reducing agents, and sea

water. It's readily welded and machined, too.

You'll do well to check the advantages of high-nickel alloys when designing for severe environments. Whether you're handling halogens, acidic or caustic corrosives, temperamental foodstuffs . . . whether temperatures are high or low, you'll find a nickel alloy to give you long, economical service.

*Registered trademark, Stainless Foundry & Engineering, Inc.

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67 Wall Street  New York 5, N. Y.

INCO NICKEL

NICKEL MAKES ALLOYS PERFORM BETTER LONGER



GENERAL NEWS

Titanium Is Improved by Noble Metal Addition

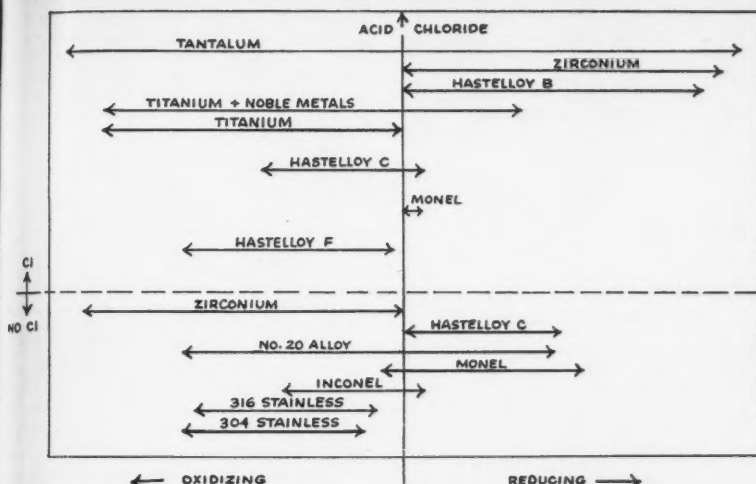


Figure 1—Comparison of titanium and titanium-noble metal alloys with other corrosion-resistant materials. The total area shown represents a variety of different types of corrosive environments. The vertical line in the center separates oxidizing environments on the left from reducing environments on the right. In addition, as one moves further and further to the left or to the right, the environment becomes

more oxidizing or reducing and more aggressive. For example, this represents an increase in temperature and concentration. Also, moving from bottom to top, the environments show greater tendency to break down passivity illustrated by an increase in acid-chloride content. The solid lines with arrows at the end show the behavior of particular alloys. Each alloy will handle all environments below its solid line.

Palladium Increases Resistance to Reducing Acids

Addition of as little as 0.1 percent palladium to titanium makes an alloy which is resistant to some boiling solutions of reducing acids, such as hydrochloric acid. The same improvement can be effected by the addition of small percentages of other metals—platinum, rhenium, ruthenium, iridium, osmium and rhodium.

The addition of 0.1 percent palladium adds about 28 cents a pound to the price of titanium, which now sells in the range \$4-14 per pound for mill products.

Tests of the mechanical properties of the titanium-palladium alloy show it is identical with properties of the unalloyed metal.

Theory Is Advanced

Development of the new alloy followed the advancement of an electrochemical theory* by Dr. Milton Stern on the role of noble metal additions on corrosion passivity which was applied in the invention. A noble metal is essentially insoluble in the corrosive environment and has a high exchange current for the hydrogen ion reduction process. When added to a metal such as titanium, a noble metal appears at the surface of the alloy to create a bi-electrode or galvanic couple and serve as a site with a low hydrogen overvoltage. This results in passivity and a marked decrease in the rate of corrosion. The effect is all the more striking when it is realized that it is produced by only one atom of palladium for every 2000 atoms in the alloy.

Since in this case, Dr. Stern's theory preceded, and led to, the discovery, it may be worth while to examine the testing procedures which resulted in the discovering and confirmation of the theory.

In a previous work, Dr. Stern had proposed that passivating-type inhibitors function primarily by creating a mixed potential more noble than the critical potential for passivity of the metal involved. The establishment of a potential more noble than some critical value is a basic concept which applies not only to inhibitors but also to the phenomenon of anodic protection and the prevention of corrosion by galvanic coupling to suitable cathode materials.

Passivity Is Established

Titanium exhibits unusually active values of critical anodic potential. Often, this potential is even more active than the reversible hydrogen potential of the solution. Thus the theory postulates the

* Metals Research Laboratories, Union Carbide Metals Co., Div. Union Carbide Corp., New York.

(Continued on Page 82)

Research Journal To Be Published In Four Sections

"Journal of Research" issued by the National Bureau of Standards will be published in four separate sections beginning July 1. To permit more effective dissemination of the NBS findings to science and industry and to broaden the journal's editorial scope, the change will divide the journal into four sections: physics and chemistry, mathematics and mathematical physics, engineering and instrumentation, and radio propagation. Readers will need to subscribe only to those sections that fall within their fields of interest.

The sections on physics and chemistry and the one on radio propagation will be issued six times a year with domestic annual subscription at \$4.00, \$4.75 foreign. The other two sections will be published quarterly with domestic annual subscriptions at \$2.25 and foreign rates \$2.75.

Subscriptions should be sent to the nearest U. S. Department of Commerce field office or Superintendent of Documents, U. S. Government Printing Office, Washington 25, D. C.

226,800 copies of 25 NACE Technical Committee Reports were distributed in 1957. In 1958, 210,300 copies of 12 NACE Technical Committee Reports were distributed.

Fracture of Materials To Be Conference Topic

A conference on Fracture of Engineering Materials will be held August 23-25 at Rensselaer Polytechnic Institute, Troy, N. Y., by Eastern N. Y. Chapter, American Society for Metals. The program will consist of consecutive sessions on ductile, brittle and quasi-brittle materials, temper embrittlement, hydrogen embrittlement, fracture, fatigue, fracture in corrosive media and other subjects.

Conference fee of \$10 payable to "Fracture Conference" should be sent to J. H. Keeler, chairman, General Electric Research Lab., Schenectady, N. Y. On-campus dining service and overnight accommodations are available on the institute's campus.

ASTM to Hold 62nd Annual Meeting on June 21-26

ASTM's 62nd annual meeting will be held June 21-26 at Chalfonte-Haddon Hall, Atlantic City, N. J. Reports of interest to corrosion engineers which will be included at the meeting are the following:

Corrosion of iron and steel, corrosion of non-ferrous metals and alloys, electrodeposited metallic coatings, chemical resistant mortars and non-destructive testing.

American Zinc Institute Elects New Officers

R. G. Kenly was elected president of the American Zinc Institute recently. He is vice president of the New Jersey Zinc Co., New York City.

Other officers include three vice presidents: T. A. Campbell, Anaconda Sales Co., H. D. Carus, Matthiessen & Hegeler Zinc Co., and E. H. Snyder, Combined Metals Reduction Co.

G. H. LeFevre, U. S. Smelting Refining and Mining Co., was re-elected as treasurer and J. L. Kimberley as executive vice president and secretary.

Building Council to Meet

The 1959 Congress of the International Council for Building Research, Studies and Documentation will be held September 21-25 in Rotterdam, Netherlands. Topics on safety factors, design and calculation of structures, heavy concrete research problems and fundamental aspects of transmission of knowledge.

X-Ray Proceedings Printed

Proceedings of the 7th Annual Conference on Industrial Applications of X-ray Analysis held in August, 1958, are available from W. M. Mueller,

Denver Research Institute, University of Denver, Denver 10, Colo.

The 359-page volume includes 25 papers. Paper-back copies are \$7.50 per copy; cloth-bound for \$8.50.

International Galvanizing Meeting to Be in France

Arrangements are being made for the 6th International Conference on hot dip galvanizing to be held in Cannes, France, May, 1961.

Subjects for discussion will include corrosion of galvanized coatings. Papers are being solicited for presentation at the conference.

Additional information can be obtained from Zinc Development Association, secretary to the EGGA, 34 Berkeley Square, London, W. 1.

Titanium Is—

(Continued From Page 81)

establishment of passivity by alloying with elements having low over-voltage characteristics.

Under conditions where the metal exhibits a relatively high hydrogen over-voltage, a passive mixed potential may not be directly obtained. However, passivity can be accomplished by alloying such a metal with an element which is essentially insoluble in the environment and which exhibits a high exchange cur-

rent for the hydrogen ion reduction process. A small amount of corrosion should leave the alloy addition essentially in elemental form on the surface. The surface is now a bi-electrode, or galvanic couple, with one of the constituents presenting a low hydrogen overvoltage surface (see Figure 2). The exchange current for the hydrogen reduction reaction on titanium is very small resulting in an active mixed potential. When alloyed with metals like platinum or palladium, regions with very high exchange currents are produced on the surface creating a mixed potential in the passive potential range. This results in a markedly reduced dissolution rate.

Other Metals Effective

The effect of ten alloy additions on the corrosion rate of titanium in boiling sulfuric and hydrochloric acids is shown in Table 1. Corrosion resistance is markedly improved in many cases. Platinum, palladium, rhodium, iridium, and ruthenium produce the best results. Osmium and rhenium are intermediate in effect. Gold is beneficial only in higher concentrations, while silver and copper are detrimental. As expected, the effectiveness of the various additions is roughly in the same order as their hydrogen overvoltage. Platinum and palladium generally exhibit very low hydrogen overvoltages, whereas gold, silver, and copper may be considered the relatively high hydrogen overvoltage elements of those evaluated.

TABLE 1—Effect of Various Alloy Additions On the Corrosion Resistance of Titanium

Composition	Weight Loss in 24 Hours ¹ (mils/yr.)	
	Boiling H ₂ SO ₄	Boiling HCl
Composition	1%	3%
Titanium.....	460	242
Ti + 0.5% Pt.....	<2	3
Ti + 0.4% Pd.....	<2	<2
Ti + 0.5% Rh.....	3	<2
Ti + 0.5% Ru.....	<2	3
Ti + 0.6% Ir.....	<2	3
Ti + 0.5% Os.....	<2	3
Ti + 0.4% Re.....	9	30
Ti + 0.5% Au.....	3	9
Ti + 0.3% Ag.....	high	high
Ti + 0.4% Cu.....	660	550

¹ The possible weighing error of these tests is ± 2 mils/year.

POSITIONS WANTED AND AVAILABLE

Active and Junior NACE members and companies seeking salaried employees may run without charge two consecutive advertisements annually under this heading, not over 35 words set in 8 point type. Advertisements to other specifications will be charged for at \$10 a column inch.

Positions Wanted

B. Sc. (Chemistry), M.C.I.C.—married, sales experience industrial chemicals and industrial water corrosion control. Desires employment Western Canada. CORROSION, Box 59-20.

Over Five Years experience in all phases of cathodic protection of pipelines and storage tanks. B. S. Degree. Age 35. Supervising experience. Complete resume on request. CORROSION, Box 59-22.

Chemical graduate, this June, Worcester Poly-Tech, desires West Coast position in city with opportunity for advanced degree study. CORROSION, Box 59-19.

Positions Available

Corrosion Engineer—Opportunity of a lifetime. Progressive top rated Eastern paint manufacturer has a splendid opening for experienced corrosion prevention salesman. Prefer man with ability to manage, supervise and train salesmen in his department. Future possibilities unlimited depending on his ability. Replies treated in strict confidence. CORROSION, Box 59-23.

Sales Engineer: Splendid opportunity for qualified engineer with broad field experience in cathodic protection. Interested in sales development work on relatively new and accepted product. Location in or near Houston but would require extensive traveling principally in Southwest. Preferred age 25-35. Reply guaranteed. Send photo & resume of experience. CORROSION, Box 59-21.

Complete line of corrosion proof materials and construction services including acid and alkali proof cements, brick, linings, coatings plastic ventilation and process equipment. This is a major line and requires technically trained agents who can devote a substantial portion of their time selling to industry and architect engineers. Several protected territories available in East, South and Midwest. CORROSION, Box 59-16.

Corrosion Engineer—Immediate and long range opportunity with a new and going organization awaits a young and ambitious mechanical or chemical engineer. Position calls for minimum of two years' experience with industrial corrosion problems. Petrochemical, refinery or related experience desired. Extensive college courses in metallurgy, chemistry and corrosion looked upon with favor. For further information or for submission of specific qualifications, write.

Texas Butadiene & Chemical Corp.
P. O. Box 777
Channelview, Texas
Or Phone
Personnel Department—GL 2-7151

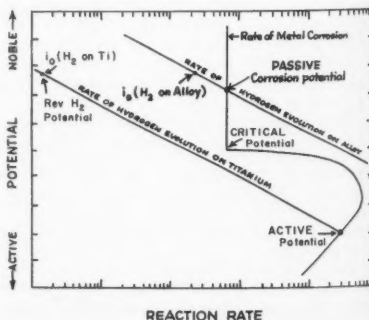
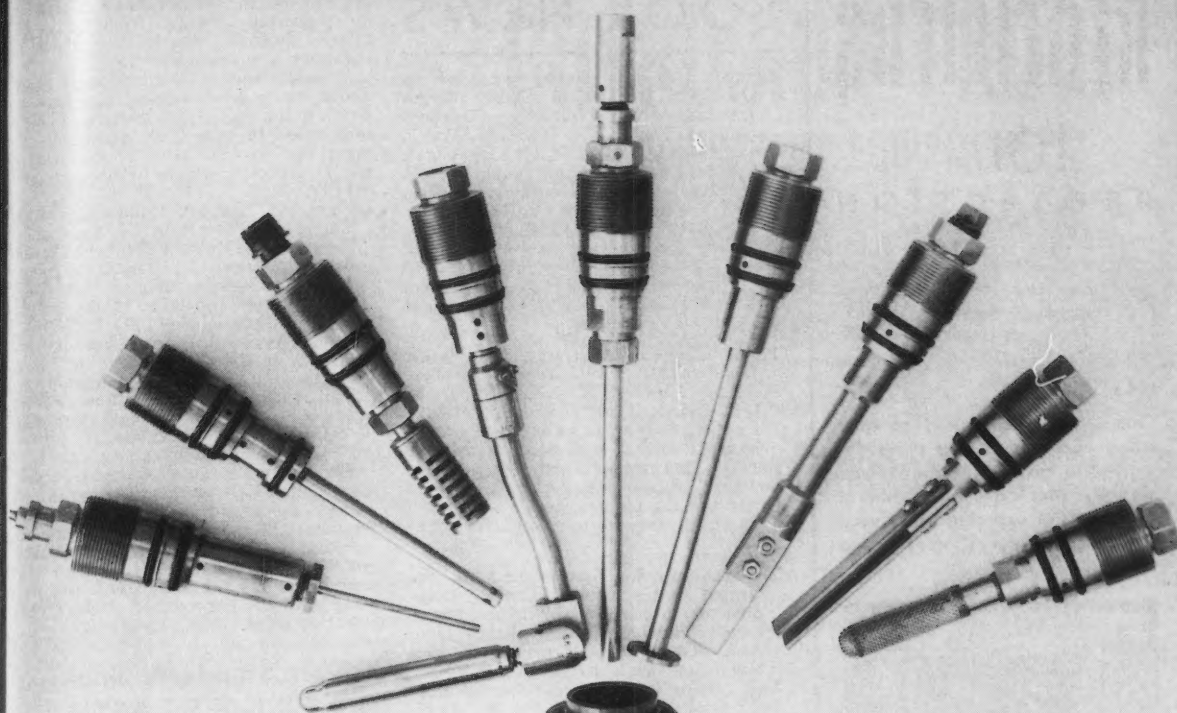


Figure 2—Schematic diagram showing how alloying with a noble metal produces a passive mixed potential and a marked reduction in corrosion rate. Alloying with palladium moves the exchange current (i_0) to the right producing a potential above the critical potential for passivity.

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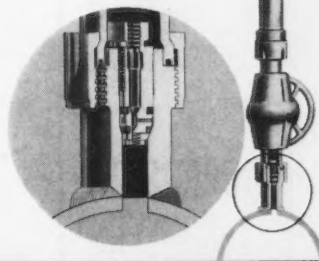
Cosasco offers the *only* complete line of corrosion survey instruments for *all* pressure conditions—up to 10,000 psi! Now you can insert and retrieve corrosion coupons, samplers, thermocouples and probes under full line pressure, without interfering with normal operations in any way. The patented Cosasco Access Fitting principle is your key to positive, accurate corrosion survey programs in transmission, storage, injection, recovery and processing equipment—at all times and under all conditions!

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Cosasco offers complete line of "use-engineered" corrosion survey instruments. In representative group shown above are (l to r): Thermocouple Survey; Product Sampler; Corrosometer* Probe; Hydrogen Probe (Chiksan swivel type); Hydrogen Probe (Standard type); Disc-type Coupon Holder (bottom of line application); Coupon Holder (Standard inside register type); Coupon Holder (Extension tongue type); Pre-Stressed Coupon Holder.

*T.M. REGISTERED

High-Pressure Retriever Assembly—Corrosion survey instruments are easily removed and replaced under full line pressure through service valve and Cosasco High-Pressure Retriever, as shown here. Access Fitting body has union type assembly with Acme outlet for fast valve installation, long thread life.



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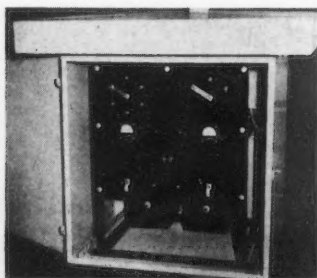
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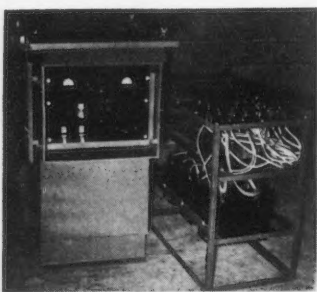
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BOOK NEWS

Identification and Determination of Organic Sulfur in Utility Gases. Institute of Gas Technology Research Bulletin No. 5. By D. McA. Mason and Henry Hakewill, Jr. 52 pages, 8½ x 11 inches, paper, January, 1959. Institute of Gas Technology, 17 West 34th St., Technology Center, Chicago 16, Ill. Price per copy, \$5.00.

Methods for accurate determination of types and amounts of organic sulfur in utility and synthesis gases are presented. Detailed drawings of special equipment are given in appendix.

Metal Finishing Abstracts. First issue, Jan-Feb 1959. Published by Robert Draper Limited, Kerbihan House, 85 Udney Park Road, Teddington, Middx., England. Annual subscription (6 issues): surface mail \$15; airmail \$20. Surveys the metal finishing literature including almost 600 periodicals of the world and the patents, books and standards of all the major industrial countries.

Technical Books Sponsored by the United States Atomic Energy Commission. 40 pages, 6 x 9 inches, paper, 1959. Available without charge from AEC Technical Information Service Extension, Oak Ridge, Tenn.

This catalog lists and describes 86 technical books published by the Commission since 1947 plus 26 volumes to be published later. Listings include contents, publishers' names and addresses, prices and indices of titles and authors.

Statistics of Extremes. By E. J. Gumbel, Adjunct Professor on Industrial Engineering, Columbia University, New York City. 347 pages, 6 x 9 inches, 1958. Columbia University Press, 2960 Broadway, New York 27, N. Y. Price per copy, \$15.00.

Considered a world authority on the subject, Dr. Gumbel has produced a reference book which may become a text for the theory and use of the distribution of extreme individuals of a sample derived from some parent population. The subject is of special interest to corrosion engineers because, for one example, the appearance of a hole in an underground pipe is a manifestation of the extreme value of the infinite array of penetrations of which the summation comprises the corrosion pit. The text assumes the reader is familiar with calculus and the elements of statistical theory.

Proceedings—National Conference on Air Pollution. 526 pages, 6 x 9, paper, 1959. P.H.S. Pub. No. 654. Public Health Service, U. S. Dept. of Health, Education and Welfare. Supt. of Documents, U. S. Government Printing Office, Washington 25 D. C. Per copy, \$1.75.

Papers and discussions presented during the Nov. 18-20, 1959 National Conference on Air Pollution are published. A wide variety of information on most of the important aspects of the air pollution problem in the United States is presented.

Symposia include: Status of knowledge, accomplishments, sources of air pollution, extent, health effects, economics and social effects, control methods and procedures, administrative aspects. Some miscellaneous discussions are in-

cluded also. Data on industrial losses are not specific. It is estimated costs of air pollution range from \$4 to 7.5 billions annually, but no breakdown of the total is provided.

Principal industrial discussion ranges about the measures being taken to reduce pollution, their cost, and their economics in terms of capital investment and payout. Most of the specific effects given pertain to health and agriculture.

Second 1957 Report. Commission 4 on Corrosion Protection of Association Belge Pour l'Etude l'Essai et l'Emploi des Matériaux. (In French) 38 Rue de Naples, Brussels, Belgium. 48 pages plus 6 pages appendix, typescript, reproduced on one side only, 8½ x 10¾, paper. Availability not indicated.

Contents include an outline of work performed, names and definitions of principal forms of deterioration of coatings applied to steel, principles followed in estimated the percentage of surface corrosion of test samples;

For 1952, a study of 67 anticorrosive paints; for 1954, comparative study of surface preparation and use of rust inhibitors and wash primers; for 1955, comparative study of anticorrosive paints and epoxy resin finishes, marine immersion and semi-immersion tests and a study of the natural corrosion of construction steel, 1954.

10-Part Standards Book Is Published by ASTM

Publication has been completed on the 10-part 1958 Book of ASTM Standards. The increase from 7 to 10 parts was caused by a growth in size of individual parts so that they could not be bound economically.

In the 10 parts are included 2450 standard specifications, methods of test, definitions of terms and recommended practices. The 10 volumes contain a total 13,600 pages.

Each part is complete with a detailed subject index and a list of standards. Individual volumes can be purchased. The complete set is priced at \$116, available from the society at 1916 Race St., Philadelphia 3, Pa.

Zinc Corrosion Research Given in Quarterly Report

Four areas of zinc corrosion research are given in the first quarterly report published April 1 jointly by the American Zinc Institute and Lead Industries Association.

The four areas are corrosion hot water storage tanks, wet storage staining of galvanized sheets, exposure of galvanized and competitive steel sheet, and zinc anode installations for marine tankers.

The report gives information on each research project: where the research is being conducted and some of the significant results.

Additional information is available from the American Zinc Institute, 60 East 42nd St., New York 17, N. Y.

Steel Founders Society

Steel Founders' Society of America has scheduled its 14th Technical and Operating Conference for November 9-11 at the Carter Hotel, Cleveland, Ohio.

New Association Promotes Aircraft Parts Castings

The Aircraft Castings Association, a non-profit corporation to promote increased use of ferrous castings for aircraft, missile and related industries, has been formed by a group of steel foundries.

Membership is open to steel foundries producing castings of aircraft quality and having the necessary facilities and personnel to advance the objectives of the association.

Additional information can be obtained by writing W. W. Stevens, Jr., Stanley Foundries, Inc., 6009 Santa Fe Ave., Huntington Park, Cal.

NACE Member Elected Head Of New Cement Association

C. E. Smith, Ceilcote Co., Cleveland, and NACE member, has been elected president of the newly formed Acid Proof Cement Manufacturers Association. Eight companies are charter members.

Located at 1015 Chestnut St., Philadelphia 7, Pa., the association proposes to handle industry statistics, advertising, new products and new uses.

Manufacturers of acid proof cements who produce a substantial portion of their requirements in their own plant, packaging and reselling under their own name are eligible for membership.

Ductile Iron Technology To Be Conference Theme

Ductile iron technology will be the subject of a one-week summer program scheduled June 29-July 3 by New York University's University Heights campus in the Bronx.

One of the 25 lectures scheduled will be "Properties and Applications of Ductile Ni-Resist" by F. G. Sefing, International Nickel Co., Inc.

Embrittlement Symposium

A symposium on hydrogen embrittlement will be included in the 1960 Convention of the American Electroplaters Society to be held July 10-14 in Los Angeles, Cal., at the Statler Hotel.

Papers for this symposium are being solicited by Herman Struckhoff, Laco, Inc., 1228 S. Atlantic, Los Angeles.

Air Pollution Meeting

The Air Pollution Control Association, 4400 Fifth Ave., Pittsburgh 13, Pa., will hold its annual meeting in Los Angeles June 22-26. Technical sessions and an exhibit will be held at the Statler Hotel.

American Welding Society

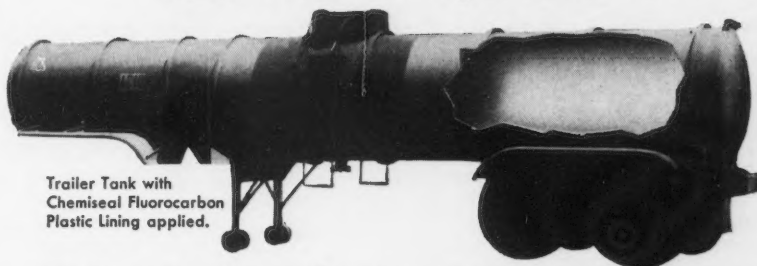
The American Welding Society's fall meeting will be in the Sheraton-Cadillac Hotel, Detroit, September 28 through October 1. Sixteen sessions will be held, and 48 technical papers presented.

The University of Illinois Corrosion Control Short Course will be held on the Urbana Campus December 7-11.

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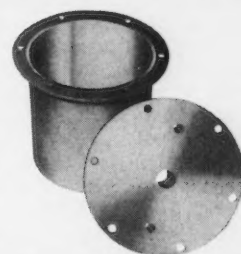
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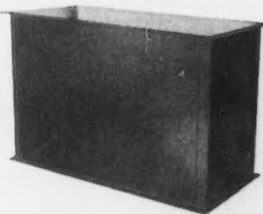
METALWELD, INC., Philadelphia, Pa.

Or, write for Bulletin AD-152, Special Products Dept., United States Gasket Company, Camden 1, New Jersey.

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ALCOA Unitrace has aluminum's natural corrosion resistance, making it well adapted for transferring molten sulfur, ammonium nitrate solutions, glacial acetic acid, fatty acids, naphthalene, phthalic anhydride, urea, wax, tar products, syrups, and numerous other products which must remain in liquid state during transfer.

*Registered Trademarks of Aluminum Company of America

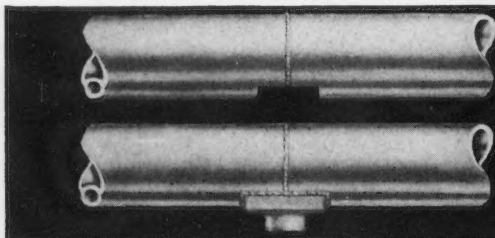
Unitrace is now available in standard pipe diameters from 1 in. through 8 in. Special cast Unitrace flanges, trace-caps, elbows and adapter flanges of ALCOA A356-T7 alloy make possible complete Unitrace piping systems.

ALCOA engineers have worked closely with all segments of the process industries for over 40 years and can help you specify the aluminum alloy tubular products and equipment best suited for your particular process applications. ALCOA's unparalleled experience in this field is available to you for the asking. Write to the address below, stating your requirements as specifically as possible. ALCOA's development engineers will welcome the opportunity to work with you on your problems.

You also can take advantage of the wide selection of free ALCOA literature on aluminum for Unitrace and other process applications. Simply check the booklets you want on the coupon and mail to the address indicated. ALCOA will forward you the material promptly and without obligation.

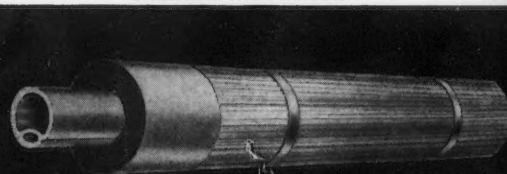
During 1959, ALCOA will conduct engineering conferences in a number of major cities on process industries applications of aluminum. Contact your nearest ALCOA sales office for full particulars and dates.

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Where flanging is not required, sections of Unitrace can be joined easily and inexpensively by employing trace-cap. This is done by cutting back the trace line about an inch on the two sections of Unitrace to be joined. The product line then can be joined with a circumferential weld using 4043 weld wire. The trace-cap is then welded over the cut-away section to seal off the steam trace section of the Unitrace.

THERMAL INSULATION

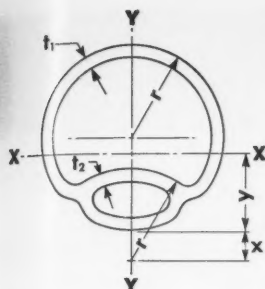


Alcoa Unitrace is designed to accommodate standard preformed pipe insulation. Boro-silicate glass wools or foams, or calcium silicate types are preferred. No protection of the aluminum surface is needed with these insulations. If cork, hair felt, asbestos cement or mineral wool is used, the aluminum surface should be coated with Aluminum Metal-and-Masonry Paint.

Temp. °F	Pressure psi	1 in.	1½ in.	2 in.	3 in.	4 in.	6 in.	8 in.
Up to 100°F	Bursting Pressure of Product Line	2980	2265	1925	1425	1210	1210	1085
	*Pressure Differential	2240	1700	1445	1070	910	910	815
	Bursting Pressure of Trace Line	2980	2265	1925	1425	1210	1210	1085
200°F	Bursting Pressure of Product Line	2800	2130	1810	1340	1140	1140	1020
	*Pressure Differential	2100	1600	1360	1005	855	855	765
	Bursting Pressure of Trace Line	2800	2130	1810	1340	1140	1140	1020
300°F	Bursting Pressure of Product Line	2530	1920	1630	1210	1030	1030	925
	*Pressure Differential	1860	1410	1200	900	755	755	675
	Bursting Pressure of Trace Line	2530	1920	1630	1210	1030	1030	925
400°F	Bursting Pressure of Product Line	1340	1020	865	650	545	545	490
	*Pressure Differential	670	510	435	320	275	275	245
	Bursting Pressure of Trace Line	1340	1020	865	650	545	545	490

*The pressure in the product line should not exceed the pressure in the trace by more than this amount.

Note: Bursting pressure data based on tests with welded Unitrace flanges and minimum mechanical properties.



Unitrace Sizes		1 in.	1½ in.	2 in.	3 in.	4 in.	6 in.	8 in.
Axis XX	Moment of Inertia (I) in. ⁴	.09	.34	.72	2.71	6.52	31.82	81.82
	Radius of Gyration (R) in.	.37	.58	.72	1.09	1.42	2.12	2.78
	Section Modulus (S) in. ³	.13	.34	.56	1.42	2.65	8.73	17.22
Axis YY	Moment of Inertia (I) in. ⁴	.09	.33	.70	2.65	6.36	29.72	76.70
	Radius of Gyration (R) in.	.37	.57	.71	1.08	1.40	2.05	2.69
	Section Modulus (S) in. ³	.14	.34	.59	1.52	2.83	8.97	17.79

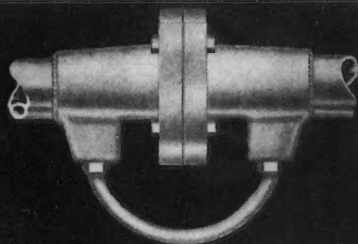
Sizes	1 in.	1½ in.	2 in.	3 in.	4 in.	6 in.	8 in.
r	.657	.950	1.187	1.750	2.250	3.312	4.312
t ₁	.133	.145	.154	.170	.187	.280	.322
t ₂	.145	.163	.174	.185	.200	.307	.354
x	.133	.344	.406	.625	.813	1.500	2.000
y	.61	.87	1.08	1.59	2.03	2.98	3.87

Unitrace Sizes	1 in.	1½ in.	2 in.	3 in.	4 in.	6 in.	8 in.
Product Area (in. ²)	.64	1.68	2.72	6.38	10.84	24.64	42.79
Trace Area (in. ²)	.10	.16	.35	.98	1.86	2.85	5.12
Metal Area (in. ²)	.65	1.03	1.39	2.28	3.24	7.05	10.60
Weight (lb/ft)	.77	1.22	1.66	2.71	3.85	8.38	12.60
*Min. Bend Radii (in.)	5	8	10½	17	24	36	48
Wetted Perimeter							
Product	3.35	4.92	6.31	9.68	12.72	18.63	24.75
Trace	1.49	1.71	2.40	4.13	5.32	6.75	9.63
Fitting Weights (lb)							
Trace-Caps	.102	.126	.240	.464	.674	1.67	2.92
Elbows	.706	1.347	2.111	5.244	9.649	27.76	52.51
Impingement Plates	.024	.039	.057	.101	.162	.22	.34
Flanges	1.186	1.919	2.851	5.515	7.762	12.93	18.62
	1-2 in.	1½-2½ in.	2-3 in.	3-4 in.	4-6 in.	6-8 in.	
Adapter Flanges	2.419	3.816	4.774	7.216	11.106	19.23	

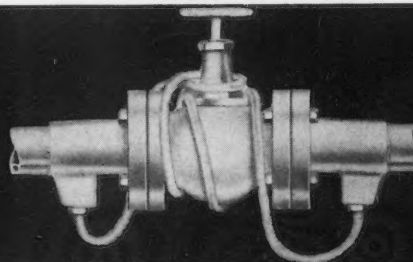
*Unitrace may be bent in any direction to these radii provided reasonable tooling is employed.

Average coefficient of thermal expansion (per °F)	-58 to +68°F	68 to 212°F	68 to 392°F	68 to 572°F
	12.1 x 10 ⁻⁶	13.0 x 10 ⁻⁶	13.6 x 10 ⁻⁶	14.2 x 10 ⁻⁶

FLANGED CONNECTIONS



VALVES AND FITTINGS



Special cast Unitrace flanges are available in alloy A356-T7. Bolt holes mate with standard 150-lb ASA pipe flanges for easy joining to other piping and equipment. For joining, the Unitrace trace line is cut back a short distance. The Unitrace is inserted into the flange and welded at the front face and neck of the flange using 4043 weld wire. Adapter flanges are available for joining different sizes of Unitrace.

Through use of Alcoa Unitrace flanges, valves and fittings can be incorporated into a Unitrace system. Tapped openings are provided in the steam chamber of Alcoa Unitrace flanges to facilitate attachment of jumper connections to traced valves and fittings. Alcoa offers Unitrace elbows with a 3x centerline radius. Several valve manufacturers offer steam-traced valves of aluminum.

Aluminum Company of America
875-F Alcoa Building, Pittsburgh 19, Pa.

Please send me the following literature on Alcoa Unitrace and other aluminum piping for the processing industry.

- ☐ 10186 Alcoa Aluminum Heat Exchanger Tubes
- ☐ 20437 Aluminum Alloy Heat Exchangers in the Process Industries
- ☐ 10460 Process Industries Applications of Alcoa Aluminum
- ☐ 10415 Welding Alcoa Aluminum
- ☐ 34-10418 Alcoa Unitrace: Combines Piping and Tracing in One Unit

Name _____

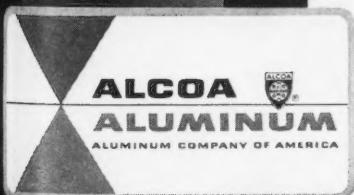
Company _____

Title _____

Address _____

City _____

State _____



For exciting drama
watch "ALCOA THEATRE,"
alternate Mondays,
NBC-TV, and
"ALCOA PRESENTS,"
every Tuesday, ABC-TV.

Equipment Services

NEW PRODUCTS

Materials Literature

Aluminum

Transit Concrete Mixers made of aluminum except for drive trains and mixer controls are being manufactured by Construction Machinery Company, Waterloo, Iowa. The mixers are built of weldable alloy 5086 developed by Kaiser Aluminum.

Bactericides

Calgon Biocide RP, a new algacide to control microbiological growths in cooling towers and evaporative condensers, has been developed by Calgon Company, P. O. Box 1346, Pittsburgh 30, Pa. This liquid inhibitor is designed to control many types of micro-organisms, algae, bacteria and fungi.

Cleaning

Ultrasonic Cleaners, manufactured by Circo Ultrasonic Corp., 50 Terminal Ave., Clark, N. J., are designed to remove excess solder flux, fingerprints, lint waxes, grease and polishing compounds from parts such as gears, bearings, instruments, etc. Each unit consists of a generator and a matching transducerized tank.

Ferox, a non-inflammable chemical rust remover, is manufactured and sold by Service Industries, 2105-07 E. Somerset St., Philadelphia 34, Pa.

Tungsten Carbide Blades for airless abrasive blast cleaning equipment are described in a bulletin available from Wheelabrator Corp., 1170 South Byrkit St., Mishawaka, Ind. The blades are designed for use in Wheelabrator abrasive blast wheels in cleaning applications with heavy sand contamination and under severe operating conditions.

Coatings—Metallic

Orosene 999, a 24k acid bright gold for electroplating, is described in a brochure available from Technic, Inc., P. O. Box 965, Providence, R. I.

Zincgrip Steel Tubing fabricated by Armco Steel Corp., 703 Curtis, Middletown, Ohio, was used for the structural members of the new microwave tower produced by Alprodco, Inc., 540 Weakley Ave., Memphis, Tenn. The new grade of tubing is made of specially treated coils of cold rolled sheet steel coated with zinc in a continuous process for corrosion protection.

Coatings—Strip

Liquid Envelope strip coating (Blue No. 67-00), supplied by Better Finishes and Coatings, Inc., Newark 5, N. J., has been sprayed on large sheets of alloy steel to protect surfaces during shipment, storage and fabrication. After fabrication, the strip coating is pulled off in big strips.

Engineering Services

Ebasco Services Inc., 2 Rector St., New York 6, N. Y., has published a brochure

describing the business consulting, engineering and construction services performed by the firm.

Fittings

Pipe-Mate, a new line of light-wall stainless steel fittings and flanges, has been introduced by Tube Turns Division of Chemetron Corp., Box 987, Louisville 1, Ky. Designed for nominal temperature, low pressure, non-critical process piping, the new line will be available in ½ to 4-inch sizes in stainless steel grades suitable for corrosive applications.

Koncentrik Pipe Unions, developed by Special Screw Products Co., Bedford, Ohio, are a line of corrosion resistant high pressure pipe unions with a confined Teflon seal. Rated from 2000 to 6000 psi, they are designed for use under extreme pressures and corrosive conditions.

Inhibitors

Nasul LP, a corrosion inhibitor for petroleum fuels, is available from R. T. Vanderbilt Co., Inc., 230 Park Ave., New York 17, N. Y. It is a non-viscous liquid designed for use in all types of petroleum fuels with physical properties that make it easy to handle, store and blend in cold weather, according to the manufacturer.

Cold Stream, a new water-soluble metalworking coolant, has been introduced by Johnson's Wax, Racine, Wis. According to the manufacturer, the coolant stops rusting, resists rancidity and reduces the threat of dermatitis.

Instruments

Cosasco Division, Perfect Circle Corp., El Monte, Cal., is producing a simplified corrosion survey instrument for taking product samples under full pressure and normal operating conditions. A modification of the firm's patented access fitting principle, the tool incorporates special design features for sealing off the top of line or vessel to permit samples being taken from the bottom or any desired position in a line.

Howell Instrument Company, 3101 Trinity St., Fort Worth 7, Texas, is producing a new turns counting dial made of light weight plastic to resist corrosion. It is designed to fit shafts of such devices as potentiometers, capacitors, valves and other equipment where micrometer read-out of settings is required. Available in a variety and combination of colors for knobs and cases, it is designated as VerniDial Model H5850.

Laboratories

Bell Laboratories is building a \$20-million research and development center at Holmdel, N. J.

Metals—Ferrous

Stainless Steel is being used by H. Paasch Marine Service Co., Erie, Pa., for special strut and rudder combinations on 52-foot boats to be used by the oil industry in South America where un-

usually corrosive condition of waters is caused by salts and chemicals.

All-Stainless Steel Buildings are being fabricated by Wonder Building Corporation of America for use in corrosive and caustic atmospheres. Available in a variety of sizes, the buildings consist of interlocking panels bolted together to form self-supporting arches that become the walls and roof.

Non-Metallics

Silicon Carbide Foam, a light weight corrosion resistant material with high porosity and thermal insulation to 4000 F, is in pilot plant production at Carborundum Company's Research and Development Division, Niagara Falls, N. Y. Possible uses will be in nuclear reactors furnaces insulation, missiles, rockets, and non-ferrous metallurgy.

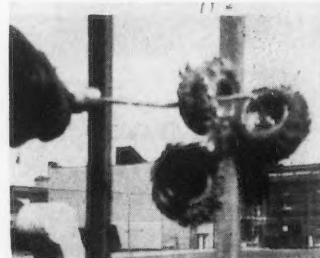
Silicon Carbide-Bonded Graphite is described in a technical data sheet available from New Products Branch, Research and Development Division, Carborundum Co., Niagara Falls, N. Y. Properties, erosion test results and fabricability are given.

Plastics

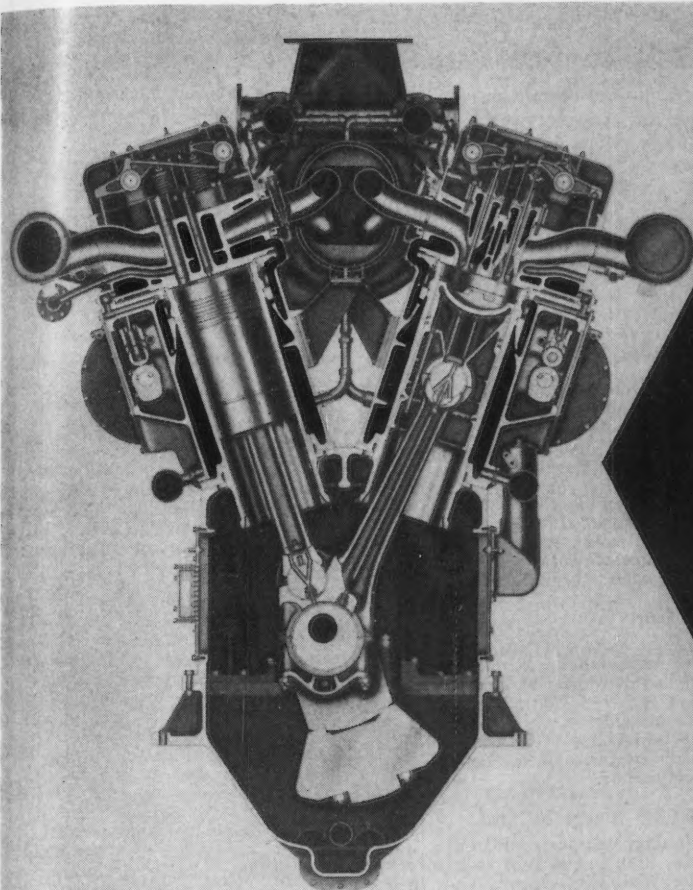
Polyethylene Piping backed by a five-year guarantee is being produced by Yardley Plastic Co., Columbus, Ohio. A new high strength polyethylene compound produced by Allied Chemical is used in the piping which has been trademarked as Golden Jet. Molecular structure of chains averaging about 70,000 carbon atoms in length has been claimed in comparison with the usual polyethylene chain of 2000 carbon atoms.

Plasta-Met is a new division of Nowery J. Smith Company, P. O. Box 7398, Houston, Texas, for the design and manufacture of plastic mechanical parts, epoxy and other resin formulations, heavy duty coatings, castings, glass fiber laminates and other plastic materials for industrial use.

(Continued on Page 90)



A new paint roller for faster painting of iron fences, pipe railings and pipe work is being produced by Speedway Paint Appliance Co., 565 55th St., Brooklyn 20, N. Y. The company claims tubular shapes can be painted in one motion and in one-quarter of the time usually required.



STOP COSTLY CORROSION

*in Diesel
cooling systems
with*

Chromate Compounds



Transverse cutaway showing part of the cooling system (solid black) in a typical 4-cycle V diesel. Compounds featuring Mutual chromates protect these vital areas.

Protecting the cooling system of a giant diesel against corrosion is just one of the jobs handled effectively and inexpensively by corrosion inhibiting compounds containing Mutual chromates. Other applications include: cooling towers, air-conditioning systems, boilers and condensers. In virtually every type of recirculating water equipment, you can protect against corrosion best with chromate compounds.

For best results, you should add a chromate inhibiting compound to your recirculating water system from the moment operations start. That way, clean metal *stays clean!* If you add them to older recirculating water systems already coated with rust and scale, they will arrest corrosion.

Compounds containing Mutual chromates offer many advantages: They are stable. They're readily soluble. They're effective against all

types of corrosion and scale forming materials. And they're available in a wide variety, each chemically tailored to a specific task. The best of them have this in common—they're based on Mutual chromates.

For the names of manufacturers of corrosion inhibiting compounds containing Mutual chromates or information on Mutual chromium chemicals, we suggest that you mail the coupon today!

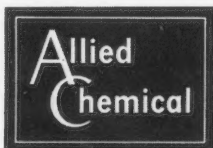
Mutual® Chromium Chemicals

Sodium Bichromate
Sodium Chromate
Chromic Acid

Potassium Bichromate
Potassium Chromate
Ammonium Bichromate

Koreon (one-bath chrome tan)

**SOLVAY PROCESS
DIVISION**
61 Broadway, New York 6, N. Y.



MUTUAL Chromium Chemicals are available through dealers and SOLVAY branch offices located in major centers from coast to coast.

SOLVAY PROCESS DIVISION
ALLIED CHEMICAL CORPORATION
61 Broadway, New York 6, N. Y.

69

Please send:

- ☐ List of manufacturers of corrosion inhibiting compounds
☐ Booklet "Mutual Chromium Chemicals"

Name _____

Position _____

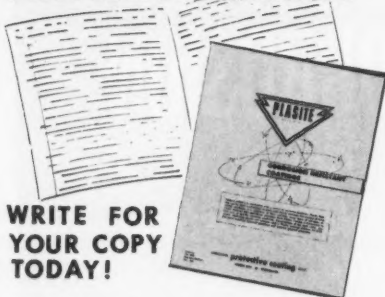
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Simplify YOUR SELECTION OF PROTECTIVE COATINGS



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TODAY!

PROVEN IN THE
PROCESS INDUSTRIES

- STEEL TANK LINING
- CONCRETE TANK LINING
- PROTECTION OF STRUCTURAL STEEL, FUME DUCTS AND EQUIPMENT
- CONCRETE FLOORS
- TANK CARS

FOR IMMERSION SERVICE
FOR HEAVY DUTY
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COLD SET COATINGS

HEAVY BUILD using standard spray or brush methods.
WIDE CHEMICAL RESISTANCE to acids, caustic solvents, salts, de-ionized water, and aqueous solutions.
HIGH TEMPERATURE RESISTANCE not affected by thermal shock.
AIR DRY field applications.
EXCELLENT BOND — No Primers on white metal blasted surface.

OTHER PLASITE PRODUCTS

INCLUDE: CAULKING COMPOUNDS
PRIMERS
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WISCONSIN
protective
coating
COMPANY GREEN BAY, WIS.
REPRESENTED IN PRINCIPAL INDUSTRIAL AREAS

PLASITE

NEW PRODUCTS

(Continued From Page 88)

Tensolite Wire and Cable insulated with Teflon FEP fluorocarbon resin has been introduced by Tensolite Insulated Wire Co., Inc., West Main St., Tarrytown, N. Y. The new insulation, according to the company, gives good weather resistance, high resistance to ozone attack, almost zero water absorption and is non-flammable.

Bondstrand Fittings for plastic pipe are being produced by Amercoat Corp., 4809 Firestone Blvd., South Gate, Cal. New fittings include 90- and 45-degree elbows, tees, couplings and other fittings for threaded-end or plain-end Bondstrand pipe. They have been tested in fresh, salt and waste water, sewage, oils, solvents, foods, beverages, gases and other corrosive solutions.

Fluoroflex-T specification details on flexible and rigid piping and accessories are given in a notebook brochure available from Resistoflex Corp., Roseland, N. J.

Publications

Electromet Metals and Alloys Review is a new magazine being published by the Electro Metallurgical Company, 30 East 42nd St., New York 17, a division of Union Carbide. It will be published quarterly and will present news on developments in ferro-alloys, metals and metal compounds.

Pumps

Proportioning Pumps for metering liquids under high pressure are being produced by Walter H. Eagan Co., Inc., 2336 Fairmont Ave., Philadelphia 30, Pa. These reciprocating plunger pumps will feed as little as 1.5 gph or as much as 108 gph of corrosive or non-corrosive liquids into chemical processes, food processes, water treatment systems and other applications where accuracy and long pump life are required, according to the company.

Milton Roy Company, 1300 E. Mermaid Lane, Philadelphia 18, Pa., is producing a controlled volume pump using the Oliver diaphragm slurry pump design principles. The new pump is designed to move corrosive and heavy concentrated slurries.

Centrifugal and Rotary Pumps for a variety of industrial uses are described and illustrated in a 72-page catalog issued by Paris Products Division, Detroit Harvester Co., 21800 Greenfield Road, Oak Park 37, Michigan. Included are Pioneer open-impeller centrifugal pumps and Rollway mechanical positive-displacement rotary pumps.

Statistics

Polyvariable Experimentation Booklet giving background information on application of statistics to experimentation is available from F. E. Satterthwaite, Statistical Engineering Institute, 8 Fuller Road, Wellesley 81, Mass. The booklet also describes four courses being offered in June on statistics.

Surfactants

Surface Active Agents Catalog has been published by Onyx Oil & Chemical Co.,

Jersey City 2, N. J. Each product is described by trade name, active ingredient, percent activity, physical state, general use, specific applications and properties. The products are divided into three general classes: anionic, cationic and non-ionic.

Tapes

Denso Products, anti-corrosion and sealing products manufactured in England, are being distributed in the United States by Denso, Inc., 480-14 Chestnut St., Camden, N. J.

Test Panels

Paint Test Panel Catalogs are available from Parker Rust Proof Co., 2177 East Milwaukee Ave., Detroit 11.

Testing Facilities

Solar Testing Service, 4165 S. W. 11th Terrace, Broward County International Airport, Fort Lauderdale, Fla., is offering testing facilities for paints, plastics and textiles in a sub-tropical atmosphere.

Titanium

Solid Tantalum Capacitors are being used in critical aircraft and missile electronics because of their insensitivity to wide and rapid temperature changes. On a testing device developed by Kemet Company, Division of Union Carbide Corp., P. O. Box 6087, Cleveland 1, Ohio, these capacitors maintained constant capacitance from -60 to +125 C. The device swings the capacitor from a vessel containing dry ice solution in trichlorethylene at about -80 C to a heated jar containing oil at about 140 C.

Valves

Luncor PVC Valves are described in a new brochure (Circular No. 601) available from Lunkenheimer Co., Cincinnati 14, Ohio. Included are tabular data on recommendations for handling various corrosive fluids, information on sizes, weights and dimensions and installation data.

Atkomatic Valve Company, 545 West Abbott St., Indianapolis, Ind., has published a new catalog listing their line of solenoid valves of bronze and stainless steel. Valve specifications, performance data and application information are given.

Steril-Aqua System for production of sterile, pyrogen free water from boiler steam is described in a bulletin available from Wilmot Castle Co., 1934 East Henrietta Road, Rochester, N. Y.

Welding

Primeweld Corporation of Dearborn, Mich., has developed a new method of fastening stainless steel moulding and appliques to automobiles by welding stainless steel fasteners to stainless steel stampings without leaving any weld marks on the pieces. Other possible uses will be in the aircraft, home appliance, furniture, radio and television industries. A patented method of welding is used in which low voltage and high amperage bond the stainless steel parts. Automobile moulding can be made and attached in three operations by the new method instead of the usual 11 separate operations.

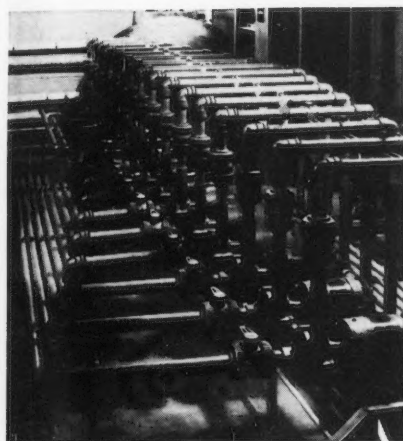
IN THE INDUSTRIAL FIELD...

NOTHING PIPES LIKE **KRALOY** PLASTIC PIPE

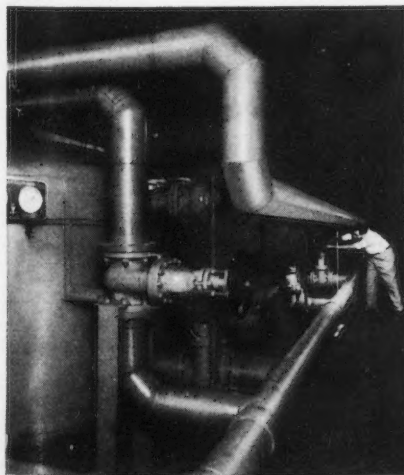
no rust, no rot, no corrosion...

no other pipe does the job so trouble-free

so permanently, at so little cost!



KRALOY PVC Plastic Pipe with plastic valves and fittings installed in the photographic laboratory at the U.S. Air Force Missile Test Center, Patrick AFB, Florida. KRALOY is unaffected by most all common acids.



Swimming pool water filters as installed at one of the leading Miami Beach hotels using 6" KRALOY PVC Plastic Pipe. Note solvent-welded joints to fit all requirements of the installation.

Industry's toughest piping problems are being solved faster, more easily and more economically than ever before by today's great trend to KRALOY Plastic Pipe. Engineers and contractors have found that KRALOY PVC Plastic Pipe meets virtually every piping need and solves corrosion and maintenance problems that no metal pipe or other materials can handle.

Unaffected by electrolysis, highly resistant to salt water scale and most all common acids, KRALOY not only avoids rust-outs and metal deterioration failures—but most important in meeting budget specifications, it is cheaper to install and maintain.

Light in weight ($\frac{1}{8}$ that of steel), KRALOY's ease of making joints and bending simplifies transportation, handling and laying—*saves up to 50% on installation costs!*

SMOOTH WALLS = HIGHER "C" FACTOR, FASTER FLOW

Quality protected by continuous laboratory control—today's KRALOY Pipe provides both normal and high impact strength. Precision extruded, KRALOY's smooth, highly polished mirror-finish walls reduce friction, permit faster, freer flow that delivers up to 20% greater volume than metal pipe... even permits use of smaller pipe sizes! And KRALOY *permanently retains* these superior flow qualities. Underground or aboveground—for liquid or use as conduit—when your requirements call for long life, corrosion and trouble-free pipe—specify KRALOY PVC Plastic Pipe. Available in all sizes from $\frac{1}{4}$ " to 12" with plastic valves and fittings.

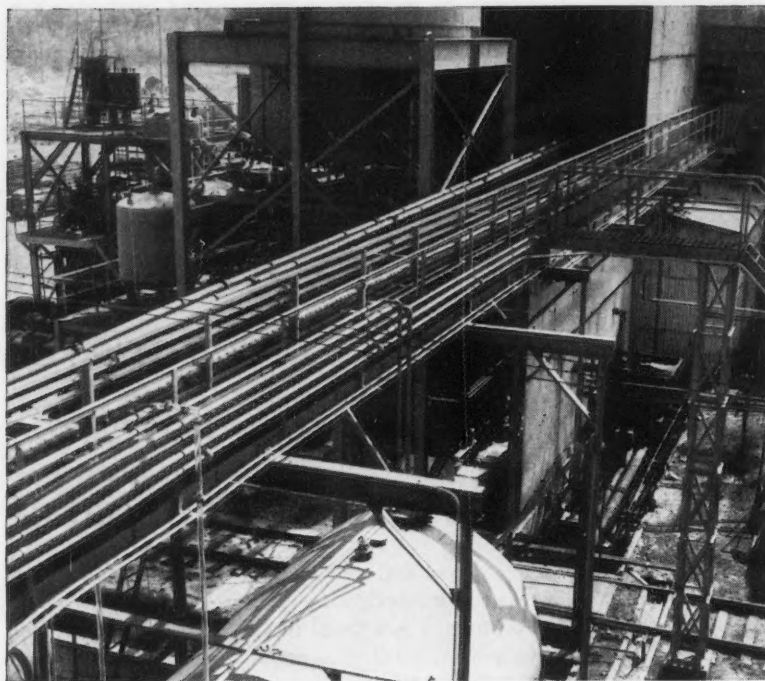
KRALOY GIVES YOU THE BIG 4

1. KRALOY is permanent—not subject to electrolysis, rot, rust or scale... needs no paint, coating, lining or cathodic protection.
2. KRALOY's flexible strength allows it to be installed faster and easier... it's easy to bend, weld and thread.
3. KRALOY's inert character and smoother walls eliminate scale which results in reduced foreign deposits in valves and fittings, giving speeded flow and cleaner delivery.
4. KRALOY's lighter weight enables 1-man handling, lets your trucks carry more pipe footage.

For complete information and technical data, write for brochure on KRALOY Plastic Pipe for Industry, Kraloy Plastic Pipe Co., Inc., Dept. C-6, 4720 East Washington Boulevard, Los Angeles 22, California.

K-59-3

NOTHING PIPES LIKE **KRALOY**



TRUSCON CHEMFAST

Combats Extreme

Corrosion Conditions!

An investment in Chemfast protection now will pay you maintenance dividends for years to come. Check these Chemfast features . . . they'll save money for you!

- Contains Devran Epoxy Resin
- Resists Chemicals
- Exceptionally durable for interior or exterior
- Resists excessive abrasion
- High Operating Temperatures — Up to 350°F.
- Available in functional, morale-building colors
- Chemical resistance effective on wood, metal and masonry surfaces
- Apply by conventional methods including hot spray



Truscon qualified representatives will provide prompt inspection, color guidance and consultation without obligation.

TRUSCON

Industrial Maintenance Division of
Devoe & Reynolds Co., Inc.



Laboratories
Detroit 11, Michigan

MEN in the NEWS

Gerald A. Baum has been added to the Resin Research Laboratories, Inc., staff as senior chemist. Ronald Saltzman is a new associate chemist with the firm.

E. L. Oliver, Jr., has joined Hodges Chemicals Co., 1661 Stierlin Road, Mountain View, Cal., as administrative vice president. R. D. Harline has been added to the staff as head of the firm's product development laboratory.

Robert C. Kany, 2651 Euston Road, Winter Park, Fla., has been appointed representative for Pennsalt Chemical Corporation's corrosion engineering products in Florida.

Three NACE members have received new positions with the Coast Paint and Lacquer Company. Carl J. Seefeld has been elected president, R. M. Johnson is now assistant to the president in charge of sales, and H. C. Owens is assistant to the president in charge of product research and development.

M. E. Merchant, senior research physicist for Cincinnati Milling Machine Co., has been presented the 1959 National Award by the American Society of Lubrication Engineers.

F. A. Ritchings has been appointed consulting mechanical engineer with Ebasco Services Inc.

Edward J. Barcal, Jr., has been named manager of the midwestern territory of the Alloy Tube Division of Carpenter Steel Co.

Harold A. Andersen, NACE member, has been appointed sales manager of Carborundum Metals Company.

Charles E. Reed has been named general manager of General Electric's metallurgical products department in Detroit.

John N. Ludwig, Jr., has been appointed supervisor of metallurgical services of International Nickel Co., Inc., Pittsburgh, Pa.

Ivan A. Miller has been named to the protective coatings sales staff of Monsanto Chemical Company's Lion Oil Division, El Dorado, Ark.

G. P. Oldham has been named vice president in charge of marketing for Allied Chemical's Barrett Division, New York 6, N. Y.

John J. Paredes has joined the sales staff of Geigy Industrial Chemicals with headquarters in St. Louis.

Joe P. Pavlich is now district engineer for Dowell Division of Dow Chemical Company at Houston.

Harold F. Schulte has been appointed chief engineer of Wheelabrator Corp., Mishawaka, Ind.

James K. Stanley and James A. Stavrolakis have been appointed section managers in technical development at Crucible Steel Company of America in Pittsburgh, Pa.

Mn

new specialty Wrought Iron offers better low temperature properties and better impact resistance than many steels

Mn (Manganese) Wrought Iron is a brand new specialty wrought iron offering excellent impact resistance at sub-zero temperatures. To define it: Mn Wrought Iron is a highly deoxidized, low carbon alloy wrought iron containing 1% manganese.

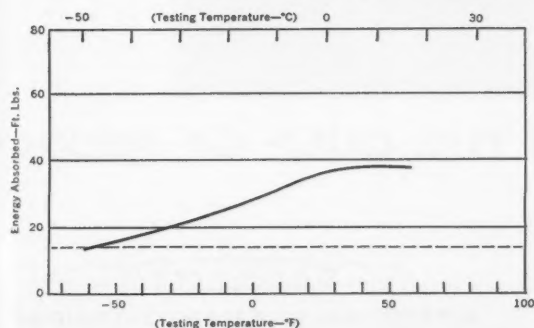
New Mn Wrought Iron is not a substitute for 4-D Wrought Iron. Mn was developed *specifically* to meet critical engineering needs for a tough, yet economical metal in low temperature services where the possibility of brittle failure poses design problems.

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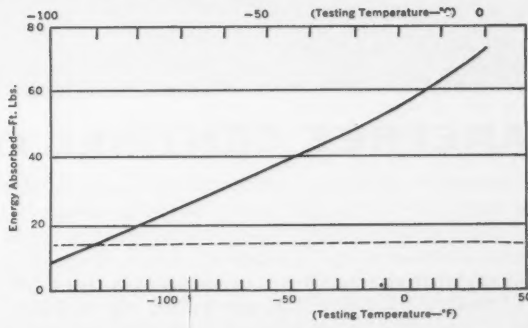
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Charpy impact values for Charpy V-notch specimens machined from conventionally-produced ½" Mn Wrought Iron plate in as-rolled condition.



Mean energy absorption: 15 ft-lbs at -58°F, notch perpendicular.



Mean energy absorption: 15 ft-lbs at -125°F, notch parallel.

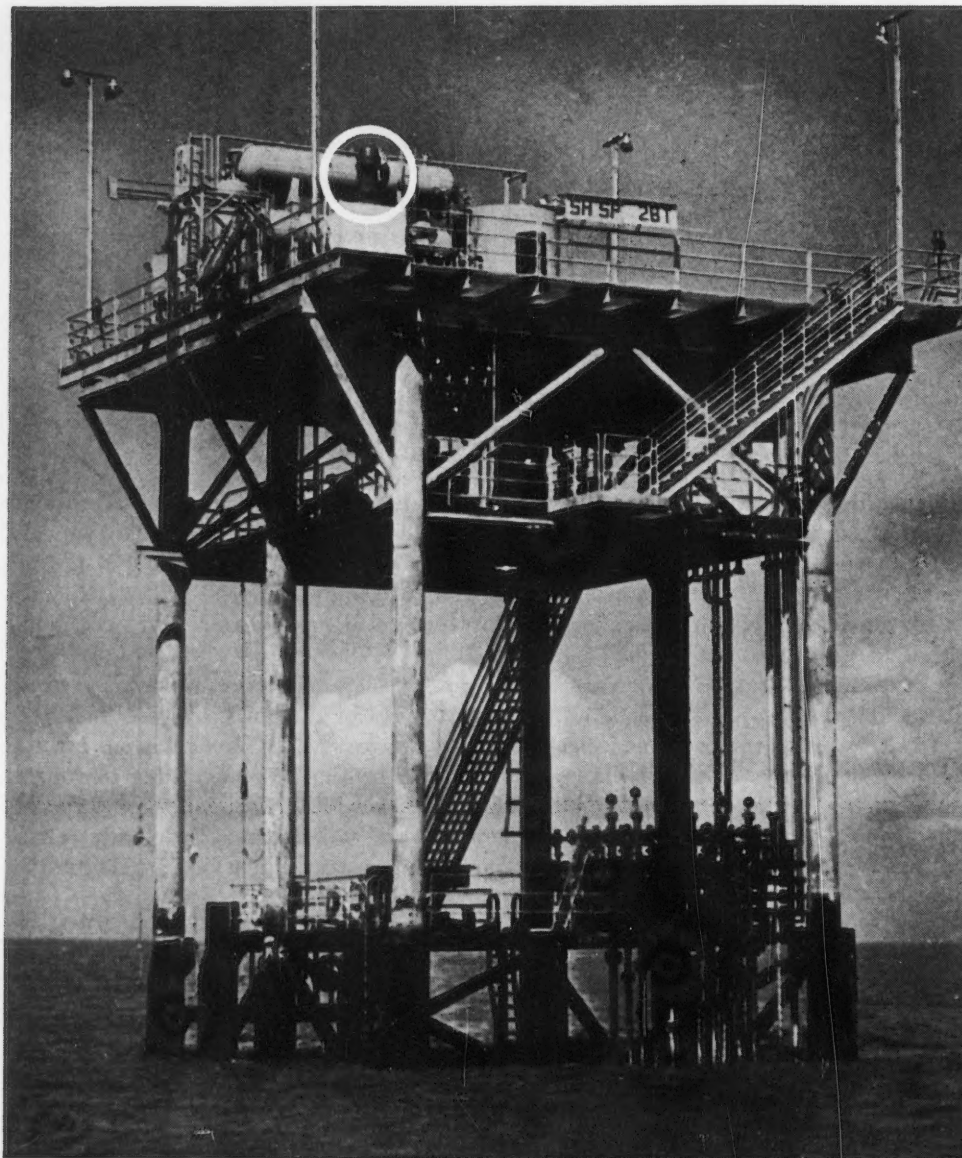


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TECHNICAL TOPICS

Solving Corrosion Problems At Electrical Generating Plants*

Introduction

AMONG THE most serious corrosion problems facing steam electric generating plants are those affecting heat exchanger tubes. The magnitude of this problem can be realized from the fact that presently operating steam generating stations of the Southern California Edison Company have 1867 tons of condenser tubes in service. At a basic price of 76 cents a pound for Admiralty tubing, this represents a replacement value of \$3,200,000.

Some of the problems encountered and solved in other parts of the company's plants are also discussed and their solutions presented. These problems involve a large number of factors including water pollution, metallurgical problems, design problems, velocities, mechanical problems created by subsidence and numerous others.

Condenser Tube Corrosion

Corrosion which necessitated retubing of condensers on a large scale became a major problem at the Long Beach Steam Station in 1944 and continued for eight years. Contributing factors were prolonged operation of the units without adequate time for cleaning or maintenance due to pressure of war demands; and severe contamination of the ocean water by industrial wastes and sewage. Oxygen content of the circulating water was near zero and hydrogen sulfide usually was present. A heavy deposit of slimy porous film in the condenser tubes allowed sulfate-reducing bacteria to cause additional attack of the tubes and it became practically impossible to operate with Admiralty alloy tubes, which had been used almost exclusively in the condensers up to that time.

The velocity of 7 feet per second was too great for Admiralty metal, condensers required retubing in less than a year, or at about 7000 service hours. Aluminum bronze, aluminum brass, 70/30 and 90/10 cupro nickel were installed in various condensers and, because of the rapidity of tube failures, the plant became a large scale field test for tubes. Extensive tests proved aluminum brass was the best answer to the problem so this alloy was adopted for all plants using ocean-circulating water.

A record was kept on the extent of corrosion in each condenser using an instrument which makes a non-destructive test of the tube wall condition.

*Revised from the paper "Corrosion Problems of an Electric Utility," by L. Baskette, Southern California Edison Company, Los Angeles, California presented at a meeting of Western Region National Association of Corrosion Engineers, Los Angeles, November 17-21, 1958.

Abstract

Some of the corrosion problems encountered at power generating plants located near salt water are considered and their solutions discussed. These include water pollution, heat exchanger tubes, condensers, concrete pipe, ground water wells, transmission tower footings, fences and structural steel. 4.6.11

Water Box Corrosion

Water boxes of condensers suffered severe graphitization. Zinc anode plates were installed but were removed later because of adverse effect on the aluminum brass tubes. Various linings and protective coatings were applied. Flame-sprayed polysulfides, polychloroprene, polyvinyl chloride paints, epoxy resins with fiberglass, polyvinyl chloride sheet linings and reinforced sprayed concrete linings have been applied.

At present accepted practice at sea-side plants for lining condenser water boxes is to be 1 1/4-inch gunite applied over reinforcement of Type 304 stainless steel 14-inch gauge wire of 2 by 2 mesh. This lining appears to be satisfactory.

Solution of one problem often creates another. Iron waste plates had to be installed to provide necessary iron in circulating water for formation of a protective iron oxide film in the aluminum brass tubes. This was because sea lines bringing in the ocean water for circulation were entirely of concrete, the joints filled with polysulfides, the screens stainless steel bronze and fiber glass, the condenser boxes concrete lined. Consequently there was no corroding iron surface presented to the incoming ocean water. The protective film that forms in the aluminum brass tube is essentially an iron oxide film, so it became necessary to install iron waste plates in the condenser water boxes, as shown in Figure 1. This was effective in stopping corrosion of the tubes.

Subsidence of Land

Because of subsidence of land in the Long Beach harbor area the maximum depth of a saucer of depression centered about the area occupied by the Long Beach Steam Station is now in excess of 24 feet. This has necessitated a number of changes at the steam station, some of which involve corrosion problems. Circulating water formerly was brought into the plant through underground concrete tunnels which had to be abandoned because of the breaks from earth movement, as well as the great depths involved. Circulating lines used now are 10-foot diameter sectionalized concrete pipe, shown in Figure 2. These are in 16-foot sections placed on saddles above ground and sealed at the

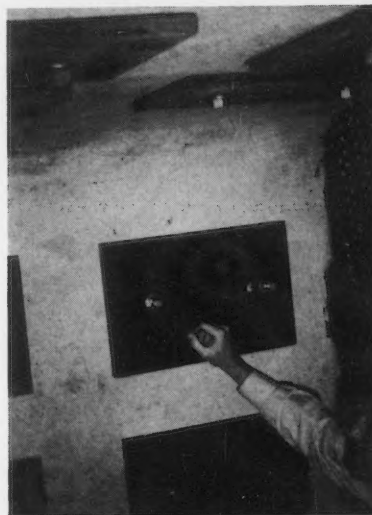


Figure 1—New waste plates and gunite lining of condenser water box.

connecting ends by means of rubber "O" rings. This method allows independent movement of the sections.

The corrosion problem on the sea line is at the "O" ring seal at the joints. The rubber "O" ring is held in position in a metal groove on the cylindrical steel end ring where the concrete sections join. Ocean water corrodes the inner rim of the groove and the vacuum inside the pipe pulls the rubber "O" ring sideways out of position, causing air leaks and possible loss of sea line siphon.

Land subsidence also has necessitated installation of a well-point system to keep the station site dry by pumping seepage water over the dyke and back to the channel. The well-points were originally 2-inch diameter copper pipe castings with fine mesh copper screens inside, set in individual sand filter beds about 20 feet below ground level. These were spaced at 10-foot intervals around the boundary to be protected and were pumped through a common header and vacuum system, thus keeping the water level several feet below ground surface. Corrosion attacked the well-points.

The copper screens completely disintegrated in a short time. The solution was to use a 6-inch asbestos-cement pipe of 20-foot length, set in a graded filter bed at the same depth and position as the original copper well-point. This pipe, closed at the bottom end, except for numerous longitudinal 1/16-inch cuts through the tube allowed water to collect to the ground water level. This water was then pulled down to a constant level by a 13-inch Hg vacuum on a one-inch plastic tube loosely set inside the transit pipe. The well-points are thus kept in operation by use of the plastic modification.

The subsidence has indirectly im-

(Continued on Page 96)



Figure 2—Sectionalized concrete pipe for circulating water at Long Beach Steam Station, Cal.

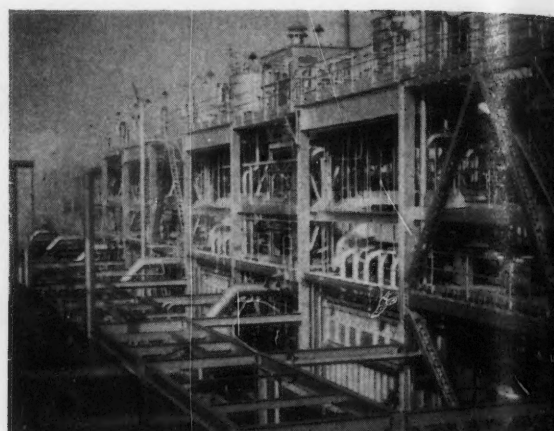


Figure 5—Exposed steel of ocean front steam station at Redondo Beach, Cal.

Solving Corrosion—

(Continued From Page 95)

proved the quality of the ocean water circulated through the condensers although elimination of sewage contamination also markedly improved this situation. Marine life has returned, con-

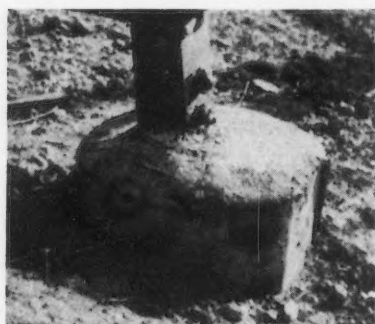


Figure 3—Concrete encasement of tower footings.

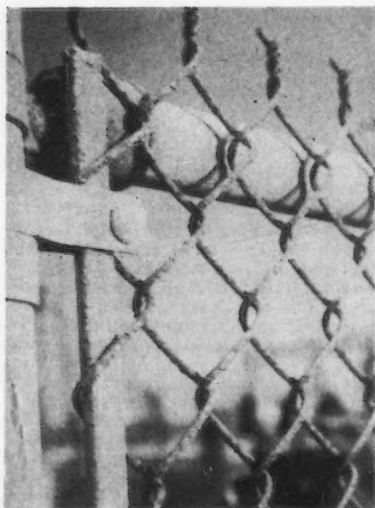


Figure 4—Close-up view of 4-year corrosion on aluminum chain link fence at ocean front exposure.

taminated circulating water has been eliminated and condenser tube life now is of the order of 80,000 service hours with no end in sight.

Sometimes it has proved more economical to bring pipe above ground and run it on saddles where frequent inspection and maintenance can be given. However, one ground corrosion problem that cannot be solved in that manner is that involving transmission tower footings.

Corrosion of Tower Footings

Old transmission lines have shown some corrosion of the steel tower footings below ground. A number of repair methods have been tried and proved ineffective, such as hot or cold applied organic coatings, zinc and magnesium anode rods and thin encasement with concrete. These methods add a few years' life but require renewal and repeated attention. The method proven most economical and permanent has been massive concrete encasement, shown in Figure 3.

This, when applied to the old all-steel footings, consists of encasing the steel and using it as reinforcement in a wide concrete block. For existing concrete footings where steel from ground line to concrete base had been exposed to a foot or more of the soil, the method is to encase this portion in a cylinder of concrete, about 18 inches in diameter, extending from the old footing to four inches above ground. Wire mesh is used to prevent concrete cracking. Cost of these repairs averages about \$19 per footing, and many thousands have been repaired. On all new lines the standard procedure is to extend the concrete footing above ground.

Corrosion of Galvanized Fence

Standard galvanized chain-link type fence is satisfactory for inland property and mountain areas, but along the seacoast, within a quarter of a mile of the ocean, it has an extremely limited life. In four years the characteristic rust-staining of galvanized wire begins when approximately 50 per cent of the zinc has corroded away. In a few more years the entire fence must be regalvanized or be allowed to continue unsightly until mechanical failure requires replacement.

Painting has not been successful and as a final recourse at tide water stations, aluminum wire fence is being used. This is aluminum alloy No. 5052-



Figure 6—Filling bolt caps with polysulfide to prevent corrosion.

H38 with wire diameter of 0.148-in and costs about \$7 a running foot. This is more than twice the cost of galvanized fence, but an extended service life is expected to justify the extra cost. After four years' service corrosion products appear to build up and the wire shows some pitting underneath. (See Figure 4.) The calculated corrosion loss is such as to reduce the wire diameter 0.5 mil per year. A grease coating on the wire, such as used on stranded high voltage aluminum conductors, undoubtedly increases the life of the wire, but might bring other complications where used on a fence.

Structural Steel Corrosion

The present trend of steam station construction is to the outdoor type with most of the structural steel exposed. Although this results in increased cost in preventing corrosion, especially at ocean front locations, considerable money is saved in wall structure.

The corrosion rate for unprotected steel at the El Segundo and Redondo Steam Station sites (shown in Figure 5) has been set at 1.2 milligrams per square inch per day, which is ten times the rate at locations 25 miles inland.

During the erection of a steam station, some rust develops on the exposed steel. After the final painting, early corrosion is noticed along edges, corners, plate contacts and bolt threads. Attainment of the desired 5 mils minimum thickness is difficult on some surfaces and coatings of alkyds, vinyls, polychloroprene, epoxy, polysulfide, coal tar and emulsions have met with varying success. One method recently developed for the protection of nuts and bolt heads has been the capping with polysulfides, as shown in Figure 6.

View showing Roskote Mastic being applied mechanically to a portion of Oklahoma Pipeline Constructors, Ltd. spread. Following minor modifications to the hot dope machine shown, an 8-man dope crew averaged 10,000 to 12,000 feet per day. Only one supply kettle was necessary, as Roskote is applied cold.



Roskote Applied Cold with Line Travel Equipment on Trans-Canada 30 Inch

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One contributing factor in the success of this construction was the adaptation of standard line travel dope machines for the easy application of Roskote cold

mastic. This combination of Roskote Mastic and modified line travel machines made possible the coating of ten to twelve thousand feet per day of 30-inch piping on the downstream side of the compressor stations.

Contractors* were able to carry on the coating operation with an 8-man dope crew, using only one supply kettle, requiring no heating fuel and thereby eliminating toxic fumes and fire hazard.

*Contractors on Trans-Canada who applied Roskote by line travel equipment:

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and Oklahoma Pipeline Constructors, Ltd.

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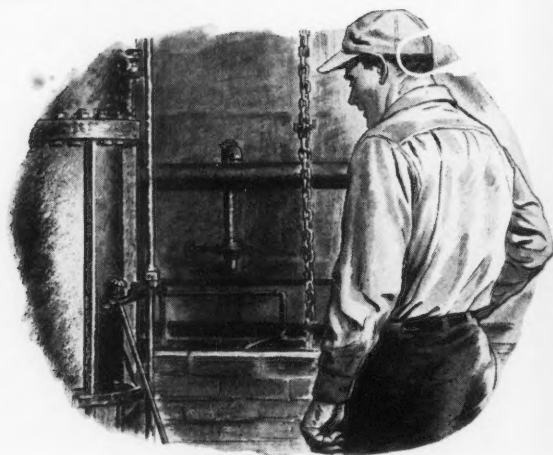
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Abstract

Corrosion problems in the production and storage of nitric acid and ammonium nitrate are discussed. Because of difference in solution potential among aluminum plates of the same nominal composition, some consideration is being given to testing each plate destined for storage tank use in 70 percent nitric acid.

Case histories include: Solution of a corrosion problem resulting from temperature differential in a tube sheet; a decision to fabricate with longer radii to avoid stress cracking of plates; stress cracking resulting from designs in which tube sheets meet Tubular Exchanger Manufacturers' Association specifications; failures from chloride containing water and zones sensitized from heating with a torch during fabrication; substitution of another analysis of aluminum for a tank bottom which failed repeatedly at the same place from what was apparently a galvanic cell; and a report on failure as a result of mercury contamination. 8.8.1

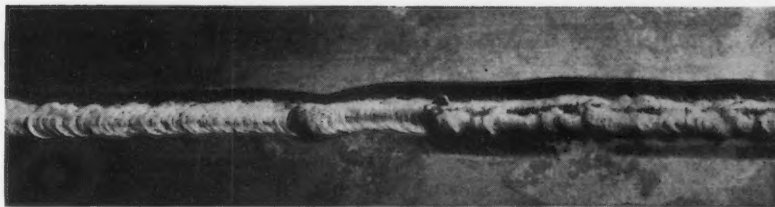


Figure 1—Corrosion on the bottom of a 5052 alloy tank that failed six times in one year. The corrosion developed as a groove adjacent to but not in the weld metal. The bottom was replaced with Type 3003 alloy with no failures during nine months of operation in the same cycles that produced the repeated failures with the 5052 alloy.

Five Case Histories on Corrosion Problems in

Nitric Acid and Ammonium Nitrate Production and Storage*

AMMONIUM NITRATE, a product of the reaction between ammonia and nitric acid, normally is stored in stainless or aluminum tanks. It usually is handled in 83 and 96 percent solutions. Of these the 96 percent gives little trouble, partly because its crystallization temperature is so high that it rarely is stored in any greater quantity than is necessary and partly because its temperature requires the use of stainless, which is substantially inert to it. On the other hand, 83 percent solutions give considerable trouble. They frequently are stored in aluminum tanks. There seems to be a wide variation in the experience with tanks of this material.

Some consideration has been given to testing each plate that goes into tanks for this service to determine if they will develop differences in potential when immersed in a violent reagent such as 70 percent nitric acid. Until the stability of aluminum for this service can be forecast, it may be necessary to use stainless steel.

Metals most frequently used in the nitric acid and ammonium nitrate industries are Type 430 stainless steel (over 15 percent chromium and low carbon) or Type 304 (either in the low carbon range or stabilized with columbium or titanium). Because most of the metals used by the author's company come from Europe, where low carbons are not made, 304 is usually replaced by Type 321. This is satisfactory, but occasionally some must be discarded because they do not pass the necessary corrosion tests (ASTM-A-262-55T). The Type 430 used is essentially the same as that made in the United States and gives no trouble in meeting specifications.

Most of the acid made and used is in the 57 to 58 percent range, but more corrosion results from condensates at about 5 percent concentration which is permitted to collect and then re-evaporate.

Case Histories of Corrosion Problems

A tube sheet with the top half cool and the bottom half heated to approximately 900 F so that acid condenses in the top sector and runs down to the bottom, has a corrosion zone in the area

where the temperature of the metal is above the boiling point of acid. Both 430 and 321 have been used with the same results. This has been corrected as far as can be determined by a false tube sheet in front of the cool half to keep it warm. The false tube sheet may be made of 304L, 321 or 347 since they do not need heat treatment. The stub tubes of the false tube sheet support it, and the spacing between it and the main tube sheet is about 1 inch. The corrosion problem is solved by keeping the metal above the dew point (about 250 F), so that condensation is prevented.

Cracking from repeated heating and cooling is another major problem. Several curved plates operating in the 800 to 1000 F zone usually crack if fabricated with short bends. In most cases the cracks are near the welds. Some cracks apparently start well out in the metal, go through the weld, follow along the other side and then come back through the weld. This sort of damage has been eliminated by using long radii (6 to 8 inches) on all bends and by carefully avoiding restraints such as reinforcing pads or attachments near the bends.

Tube sheets that operate near the 1000 F metal temperature give considerable trouble because, when made as thick as the Tubular Exchanger Manufacturers' Association requires, they tend to crack from heat stresses. On the other hand, when the tube sheet is made thinner, the thrust of the tubes creates stresses that cause cracks near the periphery of the sheets. A solution, which worries many but which usually is accepted because it will work, is a compromise in thickness between the TEMA specification and one susceptible to peripheral cracks. This is about 1 inch thick for a tube sheet 30 inches in diameter.

Tube sheets on return bend heat exchangers give trouble because of internal stresses set up when one half is in the 100 F zone while the other half in

the 900 F zone tries to expand. The gasket problem becomes extremely critical in these instances. Ring gaskets originally used bent in all directions with sections off-set as much as 1/2 inch from their original form, sometimes in the most unexpected directions. Flexitall gaskets (spiral wound, alternate strips of stainless steel and asbestos) have solved this problem, and the false tube sheet tends to equalize the temperature and thereby equalize expansion and eliminate a part of internal stresses.

A stainless heat exchanger tube may corrode from the inside where it is in contact with water while the outside in a warm nitric acid bath is not affected. This water corrosion, which often starts under scale particles, has occurred in about three cases. In one case involving Type 430, the chloride content of the water was in the 38 to 43 ppm range.

In another plant where the water was of unknown quality but generally called bad, Type 430 tubes corroded severely. In this case, tubes of Type 300 series held up better than the 430, it was reported. Several tubes in this unit obviously had been heated with a torch so they could be bent into their proper positions. At these heat affected zones, clearly delineated by the color of the metal, the tubes corroded through from the nitric acid side.

The bottom of a 5052 alloy, 30-foot deep tank failed six times in one year, almost the maximum permitted between cleanouts and repairs. The failures were alike and localized in two plates. They developed as a groove adjacent to but not in the weld metal, as shown in Figure 1.

At first, galvanic action was suspected because the steam heating coils were made of Type 304. These coils then were insulated carefully from the body of the tank (with a resistance reading of at least 500,000 ohms), but after six weeks there were new holes in the bottom in almost the same locations. The

(Continued on Page 100)

*A revision of the paper titled "Corrosion Problems in Nitric Acid and Ammonium Nitrate Industries" by D. M. Carr, The Chemical and Industrial Corp., Cincinnati, presented at a meeting of North Central Region, National Association of Corrosion Engineers, Cincinnati, October 15-17, 1958.

Methods of Testing Characteristics of Polyethylene Jackets for Steel Pipe*

Abstract

Pilot plant tests are described on a new adhesive used with a high density polyethylene as an outer jacket on steel pipe. Tests discussed are salt-crock, salt spray, penetration and thermal shock for underfilm migration and coating damage. 2.3.7

IN ITS search for materials and methods to obtain greater adhesion between steel pipe and plastic coatings, one company made minor changes in an adhesive which the company had been using for another purpose. This hot melt pressure-sensitive adhesive was tested in a pilot plant operation.

The new adhesive was used with a high density polyethylene as an outer jacket on the pipe. High density polyethylene is tougher and has a harder surface than low density polyethylene as well as higher tensile strength and heat resistance.

After having been cleaned by grit and shotblast, the pipe passes through an adhesive applicator before extrusion of the new high density polyethylene jacket. This jacket was used in a yellow color so that the heat of summer sun would be reflected rather than absorbed as would be the case with black coatings. This reduces pipe temperatures approximately 35 to 40 F during the summer months. Because thermoplastic materials are softened by heat, this means that the yellow coating is considerably tougher at ambient temperatures than a black coating.

Tests are made as follows:

A salt-crock test is used to determine the coating's resistance to underfilm migration and to check insulation resistance after continuous immersion. This test is conducted after holidays are made in the coating to expose bare pipe. Samples are tested both as the anode and as the cathode. After various periods of time, samples are removed and the coating stripped back to visually check the ability of the undercoating material to prevent underfilm migration.

On small holes or damaged areas in the coating, the elastic adhesive material will squeeze through the coating and seal the hole. Pipe drilled with small holes through the coating has been in test over six months with no change in current drain.

Salt spray tests which consist of a standard cabinet maintained at 90 F are made of specimens intentionally damaged by cutting long slits through the coating to the metal. Observations are made weekly. A similar test of immersed samples is made to determine the ability of the adhesive to prevent underfilm migration.

Penetration tests are made using a 1/2-inch blunt rod with an effective load of 200 psi. No measurable penetration after four months at room temperature has been seen.

A thermal shock test consists of immersing a sample in water for eight hours at 110 F and then subjecting it to refrigeration for 16 hours at -10 F. Samples are normally tested through thirty cycles, then the coating is examined for damage caused by temperature changes. The ends are examined for signs of underfilm corrosion. Simulated sunlight tests are run to measure ability of the coating to stand up in sunlight.

Samples tested in a soil stress box for over eight months on a regular cycling basis show no distortion.

Discussion by Sol M. Gleser, A. M. Kinney, Inc., Cincinnati, Ohio:

What is the effect of aromatics on this coating?

Reply by H. M. McDaniel:

The polyethylene jackets would not be recommended for continuous immersion in aromatics. Such materials may cause a swelling of the polyethylene.

Corrosion Keeps Knife Sharp On Fruit Canning Machine*

Corrosion of a cutter blade on a pear preparing machine keeps a usable cutting edge on the blade. Made of ordinary 1095 spring steel hardened and drawn to about 55 Rockwell C, the cutter has a useful operation life of about 80 hours in which time it will peel approximately 240,000 pears.

At the end of its operational life, the blade will be about eight thousandths thickness—one-half its original thickness. But the blade will still be as sharp as when first installed.

Corrosion on the blade, though eventually causing replacement, maintains a satisfactory cutting edge throughout the blade's service life. A blade made of stainless steel will not maintain its edge in this manner.

Experiments are being conducted with alloys which will corrode fast enough to keep the blade sharp but which will have a lower corrosion rate than the 1095 spring steel, thus giving a longer service life to the blade.

* Extracted from a paper titled "Corrosion as it Affects Design in Food Processing Equipment" by S. H. Creed, Food Machinery and Chemical Corp., San Jose, Cal., presented at a meeting of the Western Region, National Association of Corrosion Engineers, November 17-20, 1958, Los Angeles.

Nitric Acid—

(Continued From Page 99)

bottom failed again in the same location after the coils were removed from the tank.

After the bottom was removed and replaced with Type 3003 alloy, there have been no failures during about nine months of operation in substantially the same cycles that produced the repeated failures.

Reading taken at five points at the bottom of the tank showed that at no time did the temperature get above 200 F nor the pH below 5.8. The only reasonable diagnosis is that there was enough difference in solution potential among the plates of the 5052 alloy, which contains approximately 2 1/2 percent magnesium, to create galvanic cells in the bottom of the tank. Although all of the metal was within the manufacturer's specifications, it showed large potential difference among separate plates when they were immersed in 70 percent nitric acid. Undissolved iron visible in photomicrographs appeared quite differently in the stable material from that in the material which failed. Apparently there was sufficient difference in the range of minor constituent in the metal to cause a destructive potential.

Nitrate Storage Problems

An inquiry into industry's experience with 83 percent nitrate storage revealed generally satisfactory service from 3003 or 5052 aluminum although one plant reported damage from mercury and another had some unexplained holes develop in the heads of a tank. How the mercury got into the metal is unknown. This is a major problem because even one molecule of mercury will give trouble. While various sources of contamination have been suggested, including crop dusting or insect spray, there is no generally accepted answer because each case is different.

Available evidence seems to indicate that the best tank for 83 percent nitrate is one with a bottom of the purest aluminum practical, probably 3003 with sides of 5052 because of its superior tensile strength. Several tanks of these metals have given satisfactory service over a period of years.

Aluminum tanks are suitable for ammoniated ammonium nitrate solutions. For some of these solutions, stainless has corroded badly because of the solution's high pH.

Aluminum tanks rarely hold up in ammonium nitrate when the temperature of the solution is 200 F or above, doubtless because of acidic decomposition of the ammonium nitrate.

Technical Topics Scheduled for July

Use of Plastics and Synthetic Elastomers for Underground Coatings by E. G. Partridge

Mechanism of Stress Corrosion Cracking by Hugh L. Logan

Coatings Engineers Help in Power Plant Planning by W. J. Prather

* Extracted from a paper titled "Plastic Pipe and Plastic Coated Steel Pipe" by H. M. McDaniel, Republic Steel Corp., Cleveland, Ohio, presented at a meeting of the North Central Region, National Association of Corrosion Engineers, Cincinnati, October 15-17, 1958. The paper was also presented at a meeting of the Florida-Georgia Gas Association, Orlando, Florida, October 25, 1958.

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In this operation, 2000 feet of Saran Lined Pipe and many Saran lined valves are used. Most of the pipe is outdoors, resting on brackets. Because of the high physical strength of Saran Lined Pipe, no damage results from changing tempera-

tures, or from the often-great temperature differential between the pipe's contents and the outside air. Alcan's engineers specified Saran Lined Pipe for this installation because of previous highly successful experience with it in similar installations at Alcan's five other smelters.

Whenever a dependable piping system is required, whatever the degree of corrosion or chemical activity, consider Saran Lined Pipe. Saran Lined Pipe, fittings, valves and pumps are available for systems operating from vacuum to 300 psi, from below zero to 200°F. They can be cut, fitted and modified easily in the field without special equipment. For more information, write Saran Lined Pipe Company, 2415 Burdette Avenue, Ferndale, Michigan, Dept. 2284AU6.

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Abstract

Improved performance of industrial coatings achieved by good structural design is discussed. Eleven structural design situations are given in which protection from corrosion can be helped by proper design before coating application. Coating problems created by angles, sharp edges, rough areas and crevices are presented and means of eliminating the problems from lap welds and skip welding are also discussed with a recommendation that mill scale be removed before coating is applied. 5.11

Industrial Coating Performance Improved by Good Design*

THE KEY to the life and effectiveness of a coated structure under corrosive conditions are its design and construction.

When coatings are to be used to protect the structure, design for corrosion prevention means the elimination of areas which are rough, square shapes or angles where cylindrical ones would provide the same strength. It means welding in place of riveting, continuous welds in place of skip welds, machine welds wherever possible, rounded corners, fillets and in general a structure which is smooth, flowing and streamlined.

An example of the use of design to help solve the corrosion problem is the offshore drilling platforms. The original structures were steel shapes such as I-beams, angles, channels, etc. Recently, welded pipe has been used, thus taking advantage of the cylindrical surface to eliminate edges, corners, angles, and other areas difficult to protect. This realization that corrosion prevention can start with the correct design is an important step forward. Marine atmospheres, chemical plant fumes, corrosive conditions existing in rayon plants, refinery atmospheres, coke plants and other similar corrosion producing conditions first attack the areas in these plants and structures where design for corrosion resistance has not been taken into consideration.

Protection Helped by Design

Several construction situations are given below in which proper design would help coatings in protecting the structures against corrosion. On existing structures the danger spots where corrosion can occur should be recognized by plant superintendents and engineers and should be given extra care during the application of coatings. If these danger spots are recognized, good protection can be obtained; if they are overlooked, little or no protection will be achieved by the application of even the best coating.

*Extracted from a paper titled "Structural Design and Corrosion Design vs Corrosion" by C. G. Munger, Amercoat Corp., South Gate, Cal., presented at a meeting of the North Central Region, National Association of Corrosion Engineers, Cincinnati, October 15-17, 1958.

1. Structural steel angles are always a problem when a coating is to be applied. The outside of the angle is difficult to coat because coatings tend to pull away from a point or sharp edge due to surface tension. Corrosion or anodic areas thus form on edges first. A difficult area to reach with spray or brush, such as the interior of a square angle, cause coating problems. In many cases where surface contamination remains, the high tensile strength plastic coatings after drying tend to bridge the inside angle, causing an air space between the coating and the metal. In coating, spray patterns should be pointed directly toward the edge and directly into the interior of an angle to build an excess on the edge and to make sure that an adequate amount of coating will reach the interior of the angle.

2. Sharp edges or square-cut steel should be eliminated wherever possible. Coating materials tend to be thinner at edges than on planar surfaces since coatings tend to run from an edge. If the painter brushes away from the edge, the coating is brushed off, leaving a thin area vulnerable to corrosion. The coating should be brushed toward an edge, thus dragging the coating over the edge. Spraying directly at an edge will cover it and build up extra coating. This is important because moisture and condensation tend to accumulate and run toward the edge of an angle, I-beam or similar structures. Any chemical dusts or fumes will dissolve in this moisture and accumulate at the edge, thus aggravating corrosion at this point. Edges should be double coated if possible.

3. The design specifications should stipulate complete removal of mill scale prior to the application of any coating. Though not a design problem as such, mill scale or oxide scale on the exterior of steel plate, steel shapes or pipe which have been hot rolled can cause steel to pit rapidly because mill scale is cathodic to the steel. Though overcoating helps to lengthen the life of the mill scale plate under corrosive conditions, this is only a temporary measure since moisture will pass through the coating and into black iron oxide, causing the oxide

to swell slightly and pop off or loosen from the steel surface. When this happens, coating failure proceeds rapidly and again the steel goes into solution at an exaggerated rate at these spots.

Steel need not be under continuously damp or immersion conditions for this reaction to take place between the mill scale and steel. On many steel structures which are painted before the mill scale is removed, the coating breaks because a piece of the scale has lifted from the steel. Acidic fumes exaggerate such a condition, and as soon as the break is formed, rapid corrosion occurs.

4. There are three standard rivet forms—each with its own problem when coatings are required. The countersunk rivet is the easiest to coat because it is level with the bare steel and presents only a small line around the rivet's edge which requires filling with coating. Round and cone shaped rivets may be cocked forming a crevice under one side. This crevice is difficult to coat properly and will form a focal point for corrosion as will the tip of the cone shape rivets.

All rivets and overlapping steel plates in corrosive areas should be caulked tightly. Unless thoroughly caulked, this crevice containing air, moisture or chemical solution is difficult to coat properly and will be a focal point for corrosion.

5. Although a welded joint is preferable to a bolted or riveted joint from a corrosion design standpoint, welded areas present special coating problems. Coatings flow away from weld splatter, producing thinly coated areas and forming potential anodes. Weld splatter should be removed carefully where corrosion resistance is required.

Rough welding is another problem. Rough welds should be ground smooth. Weld flux often is left along the side of a weld and is difficult to remove. Because it is a strongly alkaline hydroscopic material that absorbs moisture, weld flux will create a spot where early coating failure can be expected.

6. All unnecessary scaffolding brackets and other construction aids should be removed from steel surfaces. Brackets, hold-downs and other fabricating aids remaining on steel structures after construction is completed often are cut from the surface with acetylene torches or merely broken off with a hammer, leaving a rough spot on the surface. Such areas must be smoothed for proper coating.

7. Skip welding should not be used on structures in a corrosive atmosphere. Skip welding creates crevices which are impossible to fill or cover with a coating. Continuous welds should be made at all joints.

8. Tanks that are lap welded should be welded continuously on the inside and outside. If the lapped plates are welded on the outside only, a crevice is created on the inside lap into which moisture, corrosive gases or corrosive liquids can accumulate. This crevice is difficult to coat properly. Even in old equipment, all joints should be welded continuously if a coating is to give best results.

9. When steel trusses made of angles placed back to back are used in construction, the narrow crevice formed by the washer spacing cannot be coated

(Continued on Page 103)

Industrial Coating—

(Continued From Page 102)

effectively. Corrosive fumes penetrate this crevice causing severe corrosion. If corrosive fumes are a problem, trusses should be designed to eliminate all crevices between steel members. For existing trusses, the only practical answer is to apply a bead of mastic on each side of the crevice which can be overcoated with a plastic coating.

10. In the construction of storage tanks which have cone roofs or umbrella type roofs, there is usually a center pole with I-beam rafters extending out to the edge of the tank. The steel plate roof is laid directly on top of these I-beams. Often the steel plate roof is lapped and welded only on the exterior, leaving a deep crevice between the two lapped pieces on the inside.

Two focal points are formed where severe corrosion of the roof and the I-beam rafter can occur:

Steel roof plates are not welded ordinarily to the rafter, consequently there is an area between the roof plate and the rafter which is unprotected. If a coating is to be used, the roof should be raised by wedges and this area coated properly. If possible, a rubber or plastic cushion should be applied between the steel rafter and the roof to prevent abrasion between the two caused by roof movement.

The crevice between the lapped roof plates, the second focal point, provides a reservoir for corrosive solution. If this type of construction is necessary, the roof plates should be butt welded or welded on each side of the lap so that a continuous coating can be applied.

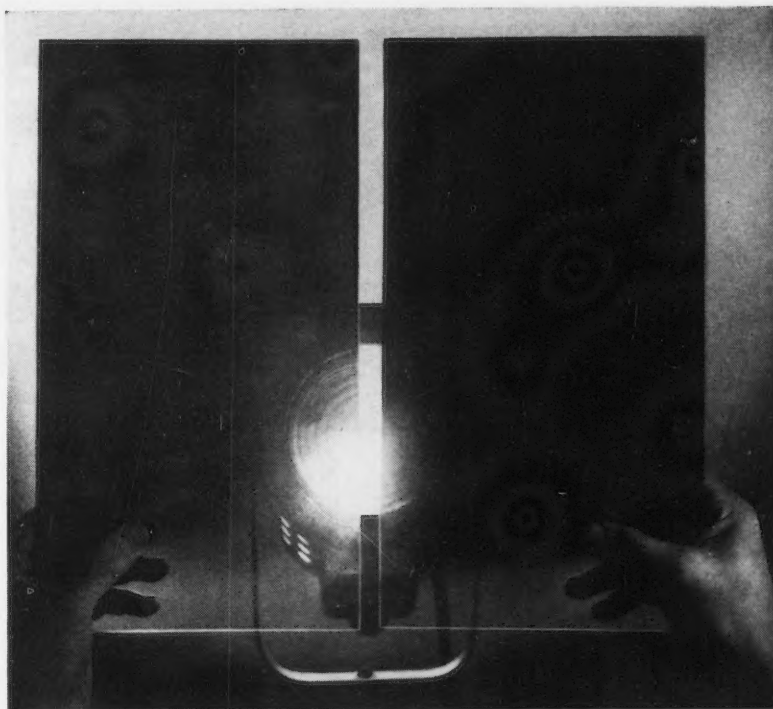
11. Piping should be used instead of I-beams or H-beams where a cylindrical cross-section is structurally sound. The amount of exposed surface area is less, and there are no edges or crevices to form potential areas of corrosion. Care must be taken also in coating pipe; the application of coating is longitudinal with the pipe, and many holidays are formed because the coating is not overlapped sufficiently during the coating process. The coating should be applied from at least four positions, and six positions are recommended to make certain that the coating is fully overlapped. Field experience has shown that pipe coating failures generally occur in a longitudinal pattern lengthwise to the pipe.

Pipe flanges, threaded joints and pipe hangers also are focal points of corrosion because crevices are formed in threaded couplings which allow the penetration of moisture and subsequent corrosion. Severe corrosion occurs at pipe hanger locations because the hanger ring usually does not fit snug enough to prevent a crevice.

Summary

Where corrosion is a problem, the design and construction of any structure should be such that as many difficult-to-coat areas as possible are eliminated. On existing buildings and equipment, these difficult areas should be given extra care during coating.

A structure which is designed with corrosion control in mind, eliminating all possible sharp edges, corners, crevices, rough welds and similar areas will operate longer, more effectively and more economically than one which is constructed in the ordinary way and then painted and repainted at short intervals for protection from corrosion.



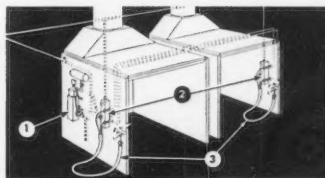
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CORROSION ABSTRACTS

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3. CHARACTERISTIC CORROSION PHENOMENA

3.2 Forms

3.2.2, 3.4.8

The Development of Hydrogen Embrittlement of Steels in Aqueous Hydrogen Sulfide Solutions. S. A. Balezin and I. V. Nikol'skii. J. Applied Chem., USSR (Zhur. Priklad. Khim.), 31, No. 8, 1181-1184 (1958).

Embrittlement is studied in hydrogen sulfide and sodium monosulfide solutions, and it is proposed that sulfur or S^{2-} reduces hydrogen overvoltage, slowing down $H^+ + H \rightarrow H_2$ recombination and thus leaving enough hydrogen atoms on the surface of the metal for diffusion. 3 tables, 24 references.—ATS. 16438

3.2.2, 3.5.8, 8.9.5

Hull Cracks on Destroyers. W. M. M. Fowden, Jr. U.S.N. J. Am. Soc. Naval Engrs., 70, No. 2, 349-353 (1958) May.

Reports occurrence of fatigue cracks in number of World War II U.S. destroyers and shows some of causes of these cracks. At Naval Ship Repair Facilities, Subic Bay, most of hull cracks were found to occur in shell plates around machinery spaces. Other cracks occurred in tank bulkhead plating and stiffeners, webs of flanges of frames, brackets and other internal structures. Fatigue type failures were prevalent. Among repairs made on hull cracks were some of stress corrosion type; specimens are shown. Slight changes in design and welding are suggested as remedies.—INCO. 16019

3.2.2, 6.4.2

Intergranular Corrosion of Aluminum-Uranium and Aluminum-Silicon-Uranium Alloys. H. C. Bowen and R. L. Dillon. General Electric Co., Han-

ford Atomic Products Operation, U. S. Atomic Energy Commission Pubn., HW-55352, March 14, 1958. 10 pp. Available from Office of Technical Services, Washington, D. C.

Aluminum-uranium and aluminum-silicon uranium alloys were tested to determine their short term resistance to intergranular corrosion in 350 C water. One-day tests were performed both on bare material and on cores canned in pre-defected Zircaloy-3 jackets. The alloys were tested in both the as-cast and wrought condition. None of the clad alloys showed any distortion when the defect in the jacket was small ($\frac{1}{16}$ in diameter). All aluminum-silicon alloys tested showed good resistance to intergranular attack in all tests; however, silicon alloys present separation problems. Cast aluminum alloys with uranium concentrations of 6 wt% or more showed resistance comparable to the aluminum-silicon alloys. (auth)—NSA. 15935

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- AWWA—Journal, American Water Works Association, Amer. Water Works Assoc., Inc., 2 Park Ave., New York 16, New York.
- BL—Current Technical Literature, Bell Telephone Laboratories, 463 West St., New York 14, New York.
- BTR—Battelle Technical Review, Battelle Memorial Institute, 505 King Ave., Columbus 1, Ohio.
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- NSA—Nuclear Science Abstracts, United States Atomic Energy Commission, Technical Information Division, Oak Ridge, Tennessee.
- OTS—Office of Technical Services, U. S. Department of Commerce, Washington 25, D. C.
- PDA—Prevention of Deterioration Abstracts, National Research Council, 2101 Constitution Ave., Washington 25, D. C.
- PMR—Platinum Metals Review, Johnson, Matthey and Co., Ltd., Hatton Garden, London, ECI.
- RML—Review of Metal Literature, American Society for Metals, 7301 Euclid Ave., Cleveland 3, Ohio.
- RPI—Review of Current Literature Relating to the Paint Colour, Varnish and Allied Industries, Research Assoc. of British Paint, Colour and Varnish Manufacturers, Paint Research Station, Waldegrave Rd., Teddington, Middlesex, England.
- SE—Stahl und Eisen, Verlag Stahl Eisen, m.b.H., Breite Strasse 27 (Schliessfach 2590), Dusseldorf, Germany.
- TIME—Transactions of The Institute of Marine Engineers, The Memorial Bldg., 76 Mark Lane, London E. C. 3, England.
- WR—Wear, Elsevier Publishing Co., 110-112 Spuistraat, Amsterdam, Holland.
- ZDA—ZDA Abstracts, Zinc Development Association, 34 Berkeley Square, London W. 1, England.

3.2.2, 3.5.8, 3.4.8

Special Steels Resistant to Stress Corrosion by Hydrogen Sulfide. P. Bastien, H. Veron and C. Roques. Paper before Société Française de Métallurgie, Paris, October 11, 1957. *Rev. Met.*, **55**, No. 4, 301-312; disc. 313-317 (1958) April.

Review of principles of embrittlement of steels. Description is given of investigations of chromium-molybdenum-vanadium steels. Results of tests on hydrogen embrittlement and on corrosion under stress showed influence of cold working, composition and mechanical properties of the steel. Steels used in tests include stainless steels and a 9.15 nickel steel. Influence of chromium content on corrosion resistance of stainless steels was studied from point of view of susceptibility to hydrogen and a scale of mechanical properties at which steels show greatest resistance to hydrogen was developed. It was found that 9 nickel steel behaves very badly and that it is impossible to anneal it at high temperature. A detailed study of the possibility of industrial heat-treatment was carried out. Graphs, photomicrographs, tables.—INCO. 16039

3.4 Chemical Effects

3.4.3, 2.3.6, 6.4.2

High Temperature Aqueous Corrosion Product Films on Aluminum. V. H. Troutner. General Electric Co., Hanford Atomic Products Operation. U. S. Atomic Energy Commission Pubn., HW-53389, Nov. 15, 1957, 39 pp. Available from Office of Technical Services, Washington, D. C.

The corrosion product films formed on aluminum alloys 1245 and X8001 in water at 310 C were examined by x-ray diffraction and by electron and optical microscopy. A technique was developed for replicating the inner surface (the surface adjacent to the base metal) of the corrosion product films for electron microscope examination. Many optical and electron micrographs and stereographs of the inner and outer corrosion product surfaces are presented. Several conclusions are reached with regard to the structure of the corrosion product film and their relationship to the corrosion processes. (auth) —NSA. 16393

3.4.3, 8.4.5

Factors Which Affect Formation and Deposition of Transport Corrosion Products in High-Temperature Recirculating Water Loops. C. Wohlberg and F. W. Kleimoia. Argonne National Lab. U. S. Atomic Energy Commission Pubn., ANL-5195 (Rev.), Dec., 1953 (Declassified Feb. 7, 1957), 78 pp. Available from Office of Technical Services, Washington, D. C.

Deposits of corrosion products form on heat transfer surfaces and in radiation flux zone at temperatures around 500 F in stainless steel systems operating with circulating water. The report considers the possible harmful effects of such deposits on heat transfer and fluid flow, as well as factors involved in the origin of these corrosion products and in the mechanisms of deposition. The prevention of deposition by chemical, mechanical and electro-static methods is discussed. (auth) —NSA. 16153

3.4.3, 4.7, 6.2.2, 6.2.5

Corrosion Products Formed in the Reaction Between Fused Sodium Hydrox-

ide and Iron-Rich Alloys of Iron, Chromium and Nickel. G. P. Smith and E. E. Hoffman. Oak Ridge National Lab. U. S. Atomic Energy Commission Pubn., ORNL-2156, May 14, 1957, 9 pp. Available from Office of Technical Services, Washington, D. C.

A study was made of the microstructures of corrosion-product layers formed by the action of fused sodium hydroxide at 815 C on types 304 and 347 stainless steel and on four high-purity iron-chromium-nickel alloys with nominal compositions of 80% iron-20% chromium, 80% iron-10% chromium-10% nickel, 74% iron-18% chromium-8% nickel and 60% iron-20% chromium-20% nickel. Each corrosion-product layer was found to consist of a nonmetallic network threading through a metallic matrix and to resemble similar layers formed by the action of hydroxide melts on Inconel. (auth) —NSA. 16280

3.4.8, 8.4.3

Hydrogen Attack of Steel in Reformer Service. A. R. Ciuffreda and Warren D. Rowland. *Proc. Am. Petroleum Inst.*, **37**, 116-128 (1957).

Case history in which three internally insulated carbon-steel catalytic reactors suffered permanent damage by hydrogen over one year. Attack varied in severity and location, depending upon metal temperature. 5 references.—MR. 16150

3.4.8, 8.4.3, 3.5.9

High Temperature Hydrogen Sulphide Corrosion in Commercial Sovaformer Units. Pt. I. E. B. Backensto, R. D. Drew and J. N. Vlachos. *Corrosion Technology*, **5**, 13-16, 21 (1958) Jan.

Hydrogen sulfide corrosion presents a problem in the platinum catalyst reforming which is used to produce the high-octane motor fuel. This corrosion can be minimized by the reduction of the hydrogen sulfide in the process stream, the use of chromium-nickel austenitic steels, and the protection of metals by aluminizing. 6 references.—MR. 16766

3.4.8, 6.2.2, 3.7.2

Sulphur Corrosion of Cast Iron. Yo. Serita. *J. Japan Foundrymen's Soc.*, **30**, 88-95 (1958) Feb. (In Japanese.)

Specimens of pure iron, iron-carbon alloy and iron-carbon with a third element (chromium, manganese, aluminum or vanadium) were heated with pure sulfur in a silica tube. Ferrite was corroded easily but cementite had good resistance. Specimens with added third element showed good resistance to corrosion except for manganese.—MR. 16630

3.5 Physical and Mechanical Effects

3.5.6, 3.7.3

The Effects of Porosity on Mild-Steel Welds. W. L. Green, M. F. Hamad and R. B. McCauley. Paper before Am. Welding Soc., Annual Spring Mtg., St. Louis, April 14-18, 1958. *Welding J.*, **37**, No. 5, 206S-209S (1958) May.

Reports study of effects of porosity on mechanical properties of mild-steel welds. Tensile, bending and impact tests were made. Butt welds were made in 1/2-in. AISI 1020 steel with welding processes adjusted to produce porosity. Test coupons were evaluated as to size or amount, shape, location and distribution of porosity. Results of tensile, bending and impact tests show that cross section of welds could be reduced by porosity up to about 7% without materially changing

mechanical properties measured. Most important factor was size or amount of porosity. Graphs.—INCO. 16475

3.5.8, 6.2.5, 4.3.6

Austenitic Chromium-Iron-Nickel Alloys Resistant to Stress Corrosion Cracking in Magnesium Chloride. H. H. UHLIG, R. A. WHITE AND J. LINCOLN, JR. Massachusetts Institute of Technology. *Acta Metallurgica*, **5**, No. 8, 473-475 (1957) Aug.

Reports on failure and resistance to failure by stress corrosion cracking mechanism, of chromium-nickel-iron stainless steels in boiling 42 percent magnesium chloride. Laboratory heats of austenitic or ferritic compositions were prepared which did not crack within 1 week or more exposure to test solution compared to cracking time of 1.5 hours for commercial Type 304. Specimens were sheared strips, stressed beyond limit to form U, insulated from apparatus, spring loaded, and fully immersed in magnesium chloride. Plastic deformation at —196 C produces more cold worked ferrite than deformation at higher temperatures; such cold worked ferrite is more crack resistant than untransformed austenite. Series of high purity 18-8's containing controlled amounts of nitrogen and carbon were tested. Those with less than 0.01 carbon and 0.01 nitrogen, did not crack within 260 hours. With 0.15 carbon or 0.15 nitrogen, cracking time was 2.5 hours and 1.2 hours, respectively. Role of nickel in providing increased resistance to stress corrosion cracking is discussed. Experiments demonstrate that precipitation process is responsible for cracking susceptibility and that austenite-to-ferrite transformation is secondary. Crack direction is dictated by bonafide carbide or nitride precipitates, or by incipient precipitates at localized concentrations of interstitial atoms. Table shows effect of carbon, nitrogen and nickel on stress corrosion cracking in magnesium chloride.—INCO. 14353

3.5.8

The Dynamic Stress Distribution Surrounding a Running Crack: A Photo-elastic Analysis. A. A. WELLS AND D. Post. U. S. Naval Research Laboratory, April, 1957, 29 pp. Available from Office of Technical Services, U. S. Dept. of Commerce, Washington 25, D. C. (Order PB 121987).

In connection with the study of brittle fractures in steel and other materials, it is of interest to observe the stress-and-strain distribution surrounding a running crack in an essentially elastic material. A photo-elastic analysis is described for the dynamic stress distribution surrounding such a crack started from one edge of a model plate of Columbia resin (CR-39) in tension. It was found that the dynamic stress distributions in the vicinity of the crack approximated static distributions in models extended at their ends by a fixed displacement. At greater distances from the crack the distributions approached those for constant load during fracture. For the crack unaffected by the presence of external plate edges, the surrounding zone of stress-and-strain disturbance grows in all directions proportionally to crack length.—OTS. 14379

3.5.8

Stress-Corrosion and Theory of Strength of Material. (In German.) KURT MATTHAES. Paper before Conference on "The Interpretation of Stress-

Corrosion of Non-Ferrous Alloys," of the Deutsche Gesellschaft für Metallkunde, Frankfurt, February, 1957. *Werkstoffe u. Korrosion*, 8, No. 5, 261-277 (1957) May.

Stress-corrosion is discussed with reference to the theory of strength. Mechanism of stress-corrosion cracking is identified with that of hot-cracking. Conditions of cracking are given. Discussion, supported by literature data, includes types of corrosion cracking, mechanical and chemical stresses, and conditions affecting tendency to cracking, simultaneous chemical and mechanical stresses, kinetics of stress-corrosion, cold deformation, condition and structure of material as function of corrosion cracking, and intercrystalline and transcrystalline cracking.—INCO. 14313

3.5.8, 6.2.5, 3.4.8

Some Case Histories of Stress Corrosion Cracking of Austenitic Stainless Steels Associated with Chlorides. H. R. COPSON and C. F. CHENG. *Corrosion*, 13, No. 6, 397-404t (1957) June.

Some 22 case histories of stress corrosion cracking in Types 302, 304, 316, 321 and 347 stainless steels are presented. These occurred in water, steam, brines and miscellaneous solutions. In each case, either the chloride content was high initially, or conditions were such that chlorides could concentrate. Usually the temperature was quite hot. Often the environment was acid and in most cases it seemed likely that air was present. Microexamination always revealed transcrystalline cracks which usually had a characteristic branching growth. In each case it was concluded that internal tensile stresses in combination with concentrated chlorides caused the cracking.

Some means of avoiding cracking are pointed out. Following the case histories the results of some laboratory tests are presented which show that resistance to cracking increases with nickel content and that large additions of nickel bring about a major improvement. Inconel, which is at the high nickel end of this series of alloys is considered immune to this type of stress corrosion cracking. 13709

3.5.8, 6.3.6

The Stress Corrosion Cracking of Copper Alloys. Rept. III. On Transcrystalline Cracking. M. SUGIYAMA AND S. UEDA. *Corrosion Engineering*, 5, No. 3, 29-32 (1956) June.

Observations were made on α/β brass. It was shown that cracks originated at the phase-boundary or the β -phase and were not observed in α -grains.

It was considered that the edge dislocations might be piled up between slip planes owing to the increment of interaction, as the effect of increase in zinc content, among the dislocations and solute atoms, or at the phase boundary as an obstacle against edge dislocations according to the following relation $\sigma_{\text{knb}} \approx \sigma_{\text{BT}}$.

The stress corrosion strain curves followed the general creep rules—JSPS. 12619

3.5.8, 6.4.2

Cumulative Fatigue Damage of Aircraft Structural Materials. Pt. 2. 2024 and 7075 Aluminum Alloy Additional Data and Evaluation. A. M. FREUDENTHAL AND R. A. HELLER. Columbia Univ. U. S. Wright Air Development Center, Tech. Note 55-273, Pt. 2, October, 1956, 22 pp.

Investigation to determine effect of randomly varying stress-amplitudes rep-

resent gust or maneuver load sequences encountered in flight on fatigue life of 7075 and 2024 aluminum alloys by testing small rotating beam specimens subject to random time series of stress-amplitudes derived from specific frequency distributions of stress-amplitudes. An approximate non-linear cumulative damage theory based on a thermal mechanism of fatigue supported by a statistical approach is developed. Theory approximates test results reasonably well for the unnotched specimens used. Graphs, tables, 12 references.—INCO. 13593



TECHNICAL REPORTS

on

Corrosion in Oil and Gas Well Equipment

T-1A Survey of Corrosion Control in California Pumping Wells. A Report of T-1A on Corrosion in Oil and Gas Well Equipment, Los Angeles Area. Pub. 54-7. Per Copy, \$5.00.

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T-1C Current Status of Corrosion Mitigation Knowledge on Sweet Oil Wells. A Report of Technical Unit Committee T-1C on Sweet Oil Well Corrosion. Per Copy, \$5.00.

T-1C Field Practices for Controlling Water Dependent Sweet Oil Well Corrosion. A Report of Technical Unit Committee T-1C on Sweet Oil Well Corrosion, Compiled by Task Group T-1C-1 on Field Practices. Pub. No. 56-3, Per Copy \$1.00.

T-1C Status of Downhole Corrosion in the East Texas Field—A Report of NACE Tech. Unit Committee T-1C on Sweet Oil Well Corrosion. Pub. 57-23, Per Copy \$5.00.

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T-1C Experience With Sweet Oil Well Tubing Coated Internally With Plastic—A Status Report of Unit Committee T-1C on Sweet Oil Well Corrosion. Pub. 58-8, Per Copy \$5.00.

TP-1D Sour Oil Well Corrosion. *Corrosion* August, 1952, issue, only at \$2.00 Per Copy.

TP-1G Field Experience With Cracking of High Strength Steel in Sour Gas and Oil Wells. (Included in Symposium on Sulfide Stress Corrosion.) (Pub. 52-3) Available only in Oct. 1952 issue of *Corrosion* at \$2.00 Per Copy.

T-1G Sulfide Corrosion Cracking of Oil Production Equipment. A Report of Technical Unit Committee T-1G on Sulfide Stress Corrosion Cracking. Pub. 54-5. Available only in Nov. 1954 Issue of *Corrosion* at \$2.00 Per Copy.

T-1G Hydrogen Absorption, Embrittlement and Fracture of Steel. A Report on Sponsored Research on Hydrogen Sulfide Stress Corrosion Cracking Carried on at Yale University, Supervised by NACE Technical Unit Committee T-1G on Sulfide Stress Corrosion Cracking by W. D. Robertson and Arnold E. Schuetz. Pub. 57-17. Per copy \$2.

T-1J Status Report of NACE Technical Unit Committee T-1J on Oil Field Structural Plastics. Per Copy \$1.00.

3.5.8, 6.3.6

Effects of Cyclic Stress and Frequency on Deformation Markings in Fatigued Copper. D. S. KEMSLEY. *J. Inst. Metals*, 85, Pt. 4, 153-157 (1956) Dec.

Fatigue deformation markings (striations) produced by cyclic stressing may be revealed on surface and in interior of copper specimens by suitable etching. Fractured rotating-cantilever specimens tested at stresses from $\pm 10,000$ to $\pm 25,000$ lb/in² and frequencies of 21 and 6000 c/m were sectioned and examined

T-1J Reports to Technical Unit Committee T-1J on Oilfield Structural Plastics. Part 1, Long-Term Creep of Pipe Extruded from Tenite Butyrate Plastics. Part 2, Structural Behavior of Unplasticized Gen Polyvinyl Chloride. Publication 55-7. Available only in June, 1955 Issue of *Corrosion* at \$2.00 Per Copy.

T-1J Summary of Data on Use of Structural Plastic Products in Oil Production. A Status Report of NACE Technical Unit Committee T-1J on Oilfield Structural Plastics. Available only in June, 1955 Issue of *Corrosion* at \$2.00 Per Copy.

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T-1M Suggested Painting Specifications for Marine Coatings. A Report of NACE Technical Unit Committee T-1M on Corrosion of Oil & Gas Well Producing Equipment in Offshore Installations. Publication No. 57-7. Per Copy \$5.00.

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by optical and electron microscopy. Striations were most numerous and distinct at low stresses and frequency in all crystals. At high stresses and frequency, striations were much less numerous and definite, appearing in only an estimated 1 crystal in 500. Evidence is given that

striations are in fact slip traces, whose characteristics change from slip packets of 1000 Å interlamellar spacing at low stress to substantially single lines at high stress. Hardness results suggest that striations are not regions of intense localized work-hardening. Localized deformation most truly characteristic of cyclic stressing occurs only in low-stress, long-life specimens and as stress increases deformation becomes less localized and exhibits similar features to those produced by unidirectional stress. Diagram, table, 18 references.—INCO. 13617

3.5.9, 4.3.6

The Effect of Temperature on the Rate of Corrosion in Metals. (In Russian.) V. V. Gerasimov and I. L. Rozenfel'd. Bull. Acad. Sci., USSR (Izvest Akad. Nauk), Chem. Sci. Section, No. 10, 1166-1171 (1957) Oct.

Behavior of iron, copper, zinc, nickel, lead and magnesium in 1.0 normal sodium chloride, 1.0 normal sodium chloride plus 0.1 normal hydrochloric acid, 1.0 normal sodium chloride plus 0.1 normal sodium hydroxide and 1.0 normal sodium chloride plus 0.1 normal KMnO solutions at 20, 50, 80 and 95 C.—BTR. 16192

3.7 Metallurgical Effects

3.7.3, 3.5.8, 5.12

Effect of Low-Temperature Stress Relieving on Stress-Corrosion Cracking. (In French.) C. R. McKinsey. Revue de la Soudure, 14, No. 1, 42-48 (1958).

Welded structures are susceptible to stress-corrosion because of the residual stresses that are generally present. A series of tests for determining the effect of low-temperature stress relieving on the stress-corrosion of arc butt welded steel plates showed that stress-corrosion increases with decrease in carbon content and that controlled relieving at low temperature reduces susceptibility of welded plates to stress-corrosion. 11 references.—MR. 16099

3.7.3, 6.2.5

Nickel-Chrome Brazing of Stainless Steel. M. E. James. Fabriform Metal Products. Western Machinery and Steel World, 49, No. 2, 68-69 (1958) Feb.

Brazing as a method of metal joining is growing in importance with the advent of supersonic flight and consequent demands for high heat and corrosion-resistant metal parts. Successful brazing of stainless steel in this jet age is now becoming more commonplace with the availability of many good heat and corrosion-resistant nickel-chromium brazing alloys, combined with furnaces which provide 100% dry hydrogen atmosphere. As corrosion resistance is a prime factor in many parts made from stainless, in the past these parts were either machined from solid stock or mechanically fabricated. Now, by nickel-chromium brazing together of such assemblies, improved parts are produced much faster and at less cost. Photos.—INCO. 15527

3.7.3, 6.2.5

Effect of High-Temperature Aging on Structure and Properties of 18% Chromium, 8% Nickel Steels. (In Russian.) Kh. I. Cheskis and S. I. Vol'fon. Metalloved. I Obrabotka Metal., No. 4, 16-25 (1958) April. Translation available from Henry Brucher, Technical Translations, P. O. Box 157, Altadena, California.

Unexplained failures of 18-8 tubes,

valves, etc. when exposed for prolonged periods to service temperatures of 500 C (930 F) and up. Study of effect of aging for up to 10,000 hours at 500-900 C (930-1650 F) on character of austenite decomposition, tensile properties, hardness and impact values of various 18-8 steels differing in their contents of carbon and such stabilizers as titanium and niobium. Steel compositions; mechanical and physical properties in various heat-treated conditions. Results of X-ray study before and after aging. Effect of carbon content, temperature and duration of aging on rate and extent of austenite decomposition and nature of resulting phases. Role of diffusional processes. 7 figures.—HB. 15637

3.7.3, 8.1.3

Uses of Oxygen and Acetylene Gases in the Refrigerator Industry. P. Bowman. Paper before International Acetylene Assoc., Annual Convention, Minneapolis April 1-3, 1957. Welding J., 37, No. 2, 120-123 (1958) Feb.

Covers many production uses of oxy-acetylene torch including welding of sheet metal, brazing, soldering and freon leak detection. All tubing joints except those to aluminum are silver brazed. A 35 silver-alloy is used for copper-to-copper, copper-to-steel, and steel-to-steel joints. Where copper tubing is silver brazed to Type 302 stainless, a 50 silver-3 nickel alloy is used to prevent possible crevice corrosion; these joints are painted with rust-resisting compound.—INCO. 15460

5. PREVENTIVE MEASURES

5.3 Metallic Coatings

5.3.2, 3.5.9, 3.5.8, 6.2.5

The Effect of Cadmium Plating on SAE 4340 Steel in the Presence of Stress Concentrations at Elevated Temperatures. E. M. Kennedy, Jr. U. S. Wright Air Development Center, U. S. Air Force. March, 1958, 40 pp. Available from Office of Technical Services, U. S. Department of Commerce, Washington 25, D. C. (Order PB 131814.)

As a result of this research, it was recommended that cadmium-plated high-strength steel not be used in applications where operating temperature approaches the melting point of cadmium. The study was conducted to determine the effect of cadmium plating on the tensile and stress rupture properties of high-strength steel at high temperatures. Service failures of cadmium-plated aircraft structural parts had indicated that stress combined with high temperature caused a sharp decrease in tensile strength. In studies of SAE 4340 steel in the annealed condition, it was shown that tensile properties are not affected by cadmium plating at 600F or 611F while under stress. Plating on the steel heat-treated to 240,000 to 260,000 psi tensile strength level causes a sharp decrease in the rupture strength at or near the melting point of cadmium (612F). However, it was observed that exposure of unstressed cadmium-plated parts to these temperatures causes no decrease in the tensile strength at room temperature. OTS. 16573

5.3.2, 6.3.15

Titanium Coating and Its Application. Pt. II. A Further Study on Titanium Cementation and Corrosion-Resistance at Normal and Elevated Temperatures. Takashi Yamaguchi and Takeshi Takei.



TECHNICAL REPORTS

on

PIPE LINE CORROSION

T-2 Statement on Minimum Requirements for Protection of Buried Pipe Lines. Prepared by a Special Task Group of NACE Technical Group Committee T-2 on Pipe Line Corrosion. Publication No. 56-15. Per Copy \$.50.

TP-2 First Interim Report on Galvanic Anode Tests. (Pub. 50-2) NACE members, \$.3; Non-members, \$.55 Per Copy.

TP-3 First Interim Report on Ground Anode Tests. (Pub. 50-1) NACE members, \$.3; Non-members, \$.55 per copy.

T-2B Final Report on Four Annual Anode Inspection Reports of Technical Unit Committee T-2B on Anodes for Impressed Current. Publication 56-1. Per Copy \$.10.

T-2B Use of High Silicon Cast Iron for Anodes. First Interim Report of Unit Committee T-2B on Anodes for Impressed Current. Publication No. 57-4. Per Copy \$.50.

T-2C Some Observations of Cathodic Protection Potential Criteria in Localized Pitting. A Report of T-2C on Minimum Current Requirements for Cathodic Protection. Pub. 54-2. Per Copy \$.50.

T-2C Some Observations on Cathodic Protection Criteria. A contribution to the work of NACE Technical Unit Committee T-2C. Publication No. 57-15. Per Copy \$.50.

T-2C Criteria for Adequate Cathodic Protection of Coated, Buried, Submerged Steel Pipelines and Similar Steel Structures. A Report of NACE Technical Unit Committee T-2C on Criteria for Cathodic Protection. Pub. 58-15. Per Copy \$.50.

T-2D Methods for Measuring Leakage Conductance of Coating on Buried or Submerged Pipe Lines—A Report of Tech. Unit Committee T-2D on Standardization of Procedures for Measuring Pipe Coating Leakage Conductance. Pub. 57-27. Per Copy \$.50.

T-2G Tentative Recommended Specifications and Practices for Coal Tar Coatings for Underground Use. A Report of Technical Unit Committee T-2G on Coal Tar Coatings for Underground Use. Per Copy \$.50.

T-2H First Interim Report. Tentative Recommended Specifications for Asphalt Type Protective Coatings for Underground Pipe Lines—Wrapped Systems. A Report of NACE Technical Unit Committee T-2H on Asphalt-type Pipe Coatings. Publication No. 57-11. Per Copy \$.50.

T-2H Second Interim Report. Tentative Recommended Specifications for Asphalt-type Protective Coatings for Underground Pipe Lines—Mastic Systems. A Report of NACE Technical Unit Committee T-2H on Asphalt-type Pipe Coatings. Publication No. 57-14. Per Copy \$.50.

T-2H Tentative Recommended Specifications for Asphalt-Type Protective Coatings for Underground Pipelines (Minimum Recommended Protection). A Report of NACE Technical Unit Committee T-2H on Asphalt Type Pipe Coatings. Pub. 58-12. 6 Copies \$.50.

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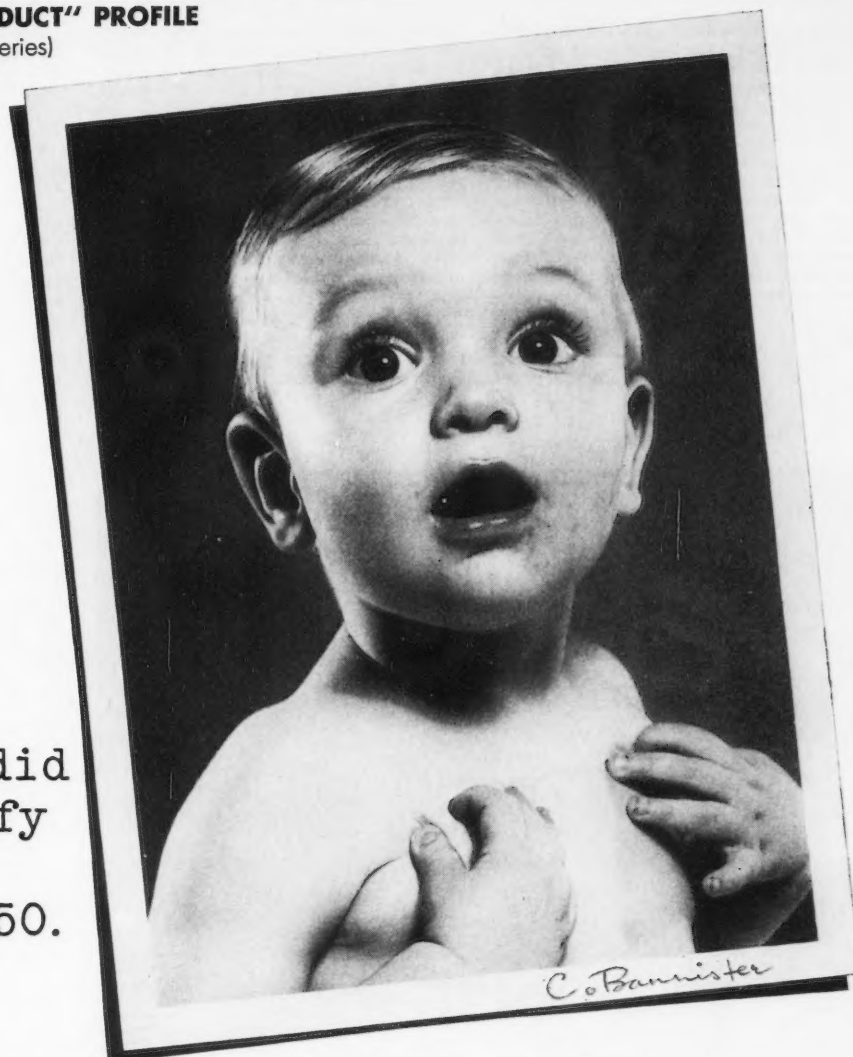
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J. Sci. Research Inst. (Tokyo), 51, 75-81 (1957) June.

Alloy coatings of titanium-containing binary alloy powders with silicon, nickel, chromium and aluminum were studied. Corrosion test and electrode-potential measurement showed that titanium-coated materials are nearly as corrosion-resistant as titanium in aqueous medium but less resistant at elevated temperatures. Scaling-resistant properties of titanium and titanium-aluminum-coated specimens, together with other resistant materials, were examined in air, hydrogen sulfide and sulfur dioxide. (auth)—NSA. 16364

5.3.4, 3.7.3

Hot-Dip Aluminum Coating on Steel. Study on Diffusion Heat-Treatment of Aluminized Steel. M. Tagaya, S. Isa and C. Ko. J. Metal Finishing Soc., Japan 9, No. 2, 41-44 (1958) Feb.

Study of the corrosion resistance of aluminized steel diffusion heat-treated at 700-1000 C for 1/2-2 hrs in the atmosphere, in solutions with 1-14 pH range, at room temperature, and at 60 C. The effect of diffusion heat-treatment on the tensile strength and the repeated bending value of the steels was also studied. A substantial growth in the diffusion layer thickness was observed when the diffusion rate was substantially increased and the base metal heated above A_{cs}, 900 C. No relationship was found between the diffusion layer thickness and the heating time.

The 700-800 C diffusion heat-treated aluminized steels were found to have lower tensile strength (<52 kg/mm²) than the hot-dip aluminum coated steels; on the contrary the aluminized steels, heat-treated at a temperature greater than 900 C, presented higher tensile

strengths (due to the remarkable growth of a hard and brittle diffused alloy layer) and decreasing repeated bending values.—JSPS. 16727

5.3.4

Research on Microthrowing Power and Leveling of Plating Baths. E. Raub. Plating, 45, No. 5, 486-492 (1958) May.

Investigations on nickel and copper plating of various macro- and micro-profiles on brass. Between macro- and microthrowing power there is a fluid transition. In copper plating from cyanide bath characteristic features of microthrowing power appear also on microprofiles if form of latter is such as to strongly repress convection of electrolyte so that thick diffusion layer forms in the depths. Leveling in realm of microthrowing power occurs with coatings from cupric sulfate and Watts nickel baths, free from addition agents, as result of definite field-oriented type of crystal growth upon metal crystallizing on sides of microprofiles. In V-shaped notches in surface positive leveling in general does not occur with random type of thickness growth. In no case does concentration of lines of current flow into depths of microprofiles occur in these baths. Genuine leveling in the sense of forcing of current lines into depths of profile occurs with bright nickel plating baths containing leveling agents. Considerably accelerated thickness growth upon lateral surfaces of microdepressions occurs. In genuine leveling field-oriented growth is observable in profile depths. But even with strongly leveling bright nickel baths growth of coating in lowest portion of profile is weaker than in middle and upper part, so that void may be formed which is covered by deposit. With brass panels provided with various shapes of notches, formation of hidden or masked cavities occur which result from increased growth of nickel deposit in upper part of microprofile. Numerous graphs, photomicrographs.—INCO. 16715

5.3.4, 3.7.4, 3.5.8

The Structure of Nickel Electrodeposits in Relation to Some Physical Properties. D. J. Evans. Trans. Faraday Soc., 54, No. 7, 1086-1091 (1958) July.

Structures of variety of nickel electrodeposits and their relationships to some physical properties were investigated, particular reference being paid to preferred orientation, internal stress, grain size, hardness, surface topography and brightness. (100) and (110) were predominant fiber axes found in matt, semi-bright and bright deposits, and for any given electrolyte (110) was found to be associated with higher values of internal stress and hardness, while (100) was associated with decreased values. Experimental evidence indicates that brightness was not due to small grain size but merely to free surface of grains being flat and parallel to general surface direction of specimen; fact that deposits also show high degree of preferred orientation is incidental and not cause of brightness. Graph, photomicrographs.—INCO. 16795

5.3.4, 6.3.9

The Use of Nickel-Aluminum Alloy Coatings for the Protection of Molybdenum from Oxidation. D. E. Couch, H. Shapiro and A. Brenner. National Bureau of Standards, J. Electrochem. Soc., 105, 485-486 (1958) August.

The protective coatings described were prepared by plating the molybdenum with 1 mil of chromium followed by 1 mils of nickel. The samples were then

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plated with 2 to 3 mils of aluminum. Several methods of forming aluminum-nickel alloy coatings were studied and the most effective method of forming the aluminum-nickel layer was by electrodeposition from a fused cryolite bath at 1000 C.—NSA. 16785

5.4 Non-Metallic Coatings and Paints

5.4.7

Mechanized Roll Coating. T. A. Dickinson. *Industrial Finishing*, 10, No. 117, 43-44 (1958).

A fast and inexpensive method of applying various types of finishing materials to sheet or plate surfaces, mechanized roll coating is used in the finishing of plywood, plastic laminates, glass, sheet metals, etc.—RPI. 16232

5.4.7, 7.1

Extend Turbine Blade Life with Metal Spray. G. C. Paraskeva and G. J. Taylor. *Power Eng.*, 61, No. 8, 68-69 (1957) August.

Metal spraying offers satisfactory solution to problem of extending life of leading edge of low-pressure stage turbine blading. An expendable metal coating is deposited which erodes instead of the blade proper. A base coating of molybdenum 0.001-0.002 in. thick is followed by a protective coating of stainless steel to the desired thickness. Technique used for metal spraying flanges is given. Photos.—INCO. 16245

5.4.7

Flame-Plated Hard Coatings. R. H. Warring. *Machinery Lloyd* (Overseas Edition), 29, No. 21, 75, 77 (1957).

With the Linde flame-plating process high wear-resistant coatings of tungsten carbide and aluminum oxide can be applied to base metals. Most experience has so far been derived from coatings of tungsten carbide-cobalt, applied by gun. The temperature of the base seldom approaches 400 F (205 C) during the process, and an applied thickness ≥ 0.010 in. is usual, with 0.002 in. minimum. The Vickers hardness of the carbide coating equals approximately 350, aluminum oxide coatings equal approximately 1000-1320, and these can be built up to 0.020 in. thick, with greatly enhanced corrosion-resistance—MA. 16368

5.4.7

Further Development in Metal-Spray Technique. Pts. III, IV. (In German.) H. Reininger. *Metalloberfläche*, 11, Nos. 10, 11; 329-333, 361-365 (1957) October, November.

An illustrated review of international developments in pistols, preliminary surface-treatments, knowledge of the structure and properties of the sprayed coatings, subsequent treatment of the coatings to improve strength and corrosion-resistant, spray-brazing and welding processes, rust and corrosion-resistant coatings, flame-sprayed bearings, repairs, finishes and testing of the finished coatings. 91 references.—MA. 16322

5.4.8

Novelties in Rust Prevention. H. Heberling. *Werkstoffe u. Korrosion*, 9, No. 3, 150-152 (1958).

Standard practice in German rust-preventive schemes is discussed with reference to removal of millscale, thin-film zinc-chrome-pigmented primers,

economic film thickness, use of fillers in primers, works priming practice and types of finishing systems.—RPI. 16504

5.4.8, 3.3.2, 8.9.5

Ship's Paints' Faults. H. Courtney Bryson. *Corrosion Prevention and Control*, 5, No. 7, 47-52 (1958) July.

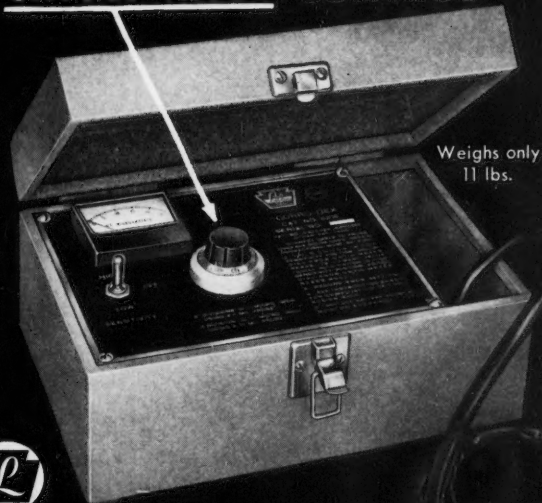
Purpose of anti-fouling paints and importance of non-fouled hulls are reviewed and factors affecting their performance enumerated. These include composition of paint, weather conditions when applied, time elapsing between application of coats and biological cycle of fouling organisms (period of year when anti-fouling paint is put onto sea). Rates of fouling differ according to length of stay in port and characteristics of ports. Leaching rates of anti-fouling paints and susceptibility of various organisms to poisons (copper and mercury ions) are discussed. Anti-corrosion paints on hull are reviewed. Importance of preventing contamination of anti-corrosion paints by copper or mercury from anti-fouling paints is stressed. Such contamination leads to formation of an electrochemically corrosive coating. Testing of anti-corrosive coat with anti-fouling paint with which it will be used is carried out by raft tests and by laboratory immersion tests.—INCO. 16747

5.4.8, 2.3.2

Antirusting Paint. A. Tarbouriech. *Ann. Inst. Tech. Batiment et Trav. Publ.*, No. 102, 571-579 (1957); *Chemical Abstracts*, 52, No. 6, 4997g (1958).

Natural and accelerated weathering do not produce the same results in evaluating antirust paint, but the latter can be standardized and used in studying paints whose resistance to water is due to the

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binder, to platemated pigment or to pigments which produce a protective chemical action.—RPI 16416

5.4.8

Rust Prevention (and Heat-Protection) with Metallic Pigments. (In German.) A. Pollack. *Metall.* 11, No. 6, 506-508 (1957).

A review of the most recent compositions and applications of rust-preventive paints pigmented with aluminum-bronze, magnesium, lead (+ lead monoxide), or zinc (+ zinc oxide) powders, and of heat-resistant paints pigmented with aluminum-bronze or zinc powders. 20 references.—MA. 16276

5.4.8, 3.7.3

Antirust Coatings and Welding. (In French and German.) R. Clement. *Z. Schweisstechn.*, 47, 212-219 (1957) Sept.

On welded constructions, the anti-rust coatings are very often altered along welds. The cause is a carbonate deposit. Means to avoid this corrosion.—MR. 16146

5.4.10

Protective Coatings for Buried Pipelines. V. M. Liss and P. Fetko. *Arabian American Oil Co. Oil and Gas J.*, 56, No. 22, 93-96 (1958) June 2.

More than 600 miles of buried pipelines were built by Aramco in Saudi Arabia. The lines pass through sandy and rocky terrain as well as through moist salt flats. Soil resistivity in these areas varies from 2,000 to 180,000 ohm-cm. Because of the low cost, local availability and generally acceptable properties, asphalt will continue to be used for coating of pipelines. Coal-tar enamel coating might be justified in areas of extremely low or widely varying soil resistivity and in oil soaked areas. Polyethylene tape appears to have many desirable properties and a seasonable installed cost. Aramco plans a full scale evaluation of this product. Photos.—INCO. 16557

5.4.10

Surface and Corrosion Protection of Underground Tubes and Cables with Plastic Wrapping. (In German.) Richard Röhm. *Tech. Mit.*, 50, 38-42 (1957) July-August.

Application of Coroplast and Corothene wrapping as protection of subterranean tubes and cables in gas works, telephone installations, petroleum industry and mining; technique of application.—MR. 16298

5.8 Inhibitors and Passivators

5.8.3, 4.3.2, 6.2.2

Behavior of Cast Iron in Acids with and without Inhibitors. (In German.) Willibald Machu and M. G. Fouad. *Werkstoffe u. Korrosion*, 9, 369-379 (1958) June.

In acids and their mixtures, effect of inhibitors is independent of the nature of their charge. Losses of weight caused by mixtures are not equal to the algebraic mean of those of the individual acids, owing to modification of hydrogen ions and anions. Organic and inorganic inhibitors behave alike. Phosphate ions of phosphoric acid act as inhibitors. Carbides and silicides in the surface of the cast iron reduce inhibition. Structure of inhibitor film as well as solubility and resistance of surface film determine degree of inhibition. 7 references.—MR. 16541

5.8.3, 2.3.5, 3.6.8

An Electrochemical Method of Testing Corrosion Inhibitors in Neutral Salt Solution. T. Kobayashi and M. Nagayama. *Corrosion Engineering (Japan)*, 7, No. 3, 25-28 (1958) May.

Polarization resistance and weight loss of mild steel were measured in 1% sodium chloride solution containing various quantity of sodium silicate as corrosion inhibitor. The effect of adding a certain kind of metal chloride to the above solution was also investigated. The corrosion rate was found to decrease as the polarization resistance increased, and their relation was linear on log-log plot. The corrosion rate, and its variation with time, can thus be deduced from the measurement of polarization resistance. The main advantage of this method lies in the fact that the specimen does not have to be taken out of the solution.

A considerable increase in the inhibiting effect of sodium silicate was noticed in the presence of either one of the zinc, manganese or nickel ions. A certain weight-ratio determining the maximum inhibiting effect was also found.—JSPS. 16571

5.8.3, 3.8.3

The Mechanism of Passivating-Type Inhibitors. Milton Stern. *J. Electrochem. Soc.*, 105, No. 11, 638-647 (1958).

All passivating-type inhibitors are oxidizing agents. However, not all oxidizing agents are inhibitors. A passivating-type inhibitor functions by producing local-action current which anodically polarizes a metal into the passive potential region and thereby provides the means for obtaining a noble mixed potential. This mechanism is independent of whether passivity is caused primarily by oxide or adsorbed films. The major factors which determine whether a particular system will exhibit passivity are: (a) reversible potential of the redox system created by the oxidizing inhibitor; (b) electrochemical parameters for reduction of the inhibitor on the metal surface (exchange current, Tafel slope, and limiting diffusion current), and (c) critical anodic current and Flade potential of the metal. If oxygen is present in the system, some chemicals may function by changing its reduction kinetics. It is further shown that the amount of inhibitor found associated with the surface is not necessarily related to adsorption. Data are presented for passivation of stainless steel and titanium. The mechanism is discussed in terms of various oxidizing agents including oxygen, chromate, molybdate, and pertechnetate. 16631

5.9 Surface Treatment

5.9.4

Applications of Bonderizing in the Field of Light Alloys. P. de Cerna. *Alluminio*, 27, No. 7-8, 337-340 (1958) July-August.

After having described the procedure for the application on aluminum and aluminum alloys of phosphate coatings which protect the metal from corrosion and ensure good adherence of paint, the advantages of various types of commercially available Bonderite products are evaluated. It is pointed out that phosphate coatings efficiently protect the metal during cold working and, in general, they help to prevent untimely deterioration of the metal caused by wear.—ALL. 16498

5.9.4, 3.4.6

Phosphoric Acid Treatments for Steel. Pt. I. Nature of Coatings Produced by the Action of Phosphoric Acid on Steel. M. Donovan, J. W. Scott and L. L. Shreir. *J. Applied Chem.*, 8, Pt. 2, 87-96 (1958).

The nature of the mixed iron phosphates present in coatings produced by the action of phosphoric acid on a clean steel surface has been investigated. The composition of the coating changes with time of exposure to atmospheric oxygen. The role of oxygen in the reaction mechanism is discussed.—RPI. 16788

5.9.4

Modern Methods of Protection of Metallic Surfaces. P. Knezevic. *Zastita Materijala*, 6, No. 3, 121-126 (1958).

The methods of phosphatization of metals are described, especially the phosphatization of iron and steel. The physical and chemical properties of the phosphatized layer are given. Foreign standards for application of this method are quoted. The second part of the paper deals with the "Hydrofinish" method of cleaning of metallic surfaces, which employs the impact of injected air in which an abrasive liquid is suspended.—RPI. 16568

5.9.4

The Selection and Use of Phosphate Coatings as Pre-Treatments for Organic Finishes. R. F. Drysdale. Paper before Inst. Metal Finishing. *Electroplating and Metal Finishing*, 11, No. 1, 2; 9-14, 51-55 (1958) Jan., Feb.

Reviews types of phosphating processes available to industry at present and discusses principles to be considered in selecting process for more widely used applications. Discussion is limited to phosphate coatings for anti-corrosion or paint bonding purposes. In analyzing characteristics of phosphate coatings, particular reference is made to relation between weight of phosphate per unit area and various properties of coating. Crystal size, free pore area, coating thickness, corrosive residues and resistance to deformation are considered. With regard to application, wetting characteristics, treatment time, plant considerations, surface cleaning and surface physical conditions are discussed. Types of processes include: heavy, medium, light and very light coating processes, phosphate passivation processes and processes for application in situ (cold treatment). Salt spray test specimens are shown.—INCO. 16495

6. MATERIALS OF CONSTRUCTION

6.2 Ferrous Metals and Alloys

6.2.5

Chromium-Aluminum Steels, Their Properties and Applications. (In French.) Eugene Herzog. *Corrosion et Anticorrosion*, 6, 117-126 (1958) April. Principal characteristics of chromium-aluminum steels. Resistance to fissuring in solutions of nitrates and in hydrogen sulfide. Surface condition and corrosion rate; transformations and mechanical properties; results of corrosion tests.—MR. 16514

6.2.4, 2.2.3, 3.7.2

The Corrosion Resistance of Low-Alloy Steels. E. Herzog. *J. Iron & Steel Inst.*, 187, Pt. 1, 46-47 (1957) Sept.

Discusses results of tests made in Inco's Kure Beach since 1948. Table



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shows variation with time of rates of corrosion of chromium and chromium-aluminum steels. Effect of aluminum is less than that of chromium, but is, nevertheless of the order of about 20%. These tests were made at a distance of about 100 m. from water's edge. Other tests show action of aluminum more clearly. Very close to water's edge, when exposed to spray, its action is very pronounced, while further away, it is less pronounced. Results show that influence of aluminum appears to depend on degree of humidity and extent to which it varies; it is only favorable when humidity is high. Results of immersion of aluminum and steel in calm sea water for three years are given. Tables, graphs.—INCO. 16175

6.2.5, 3.7.2

Study of Austenitic Chrome-Manganese Steels. A. S. Panicheva. Metalloved. I Obrabotka Metallov, No. 5, 23-27 (1958) May. Translation available from Henry Bratcher, Technical Translations, P. O. Box 157, Altadena, Calif.

Chrome-manganese austenitic steels as satisfactory replacements for chromium-nickel steels for service requiring resistance to scaling and creep. Effect of various alloying elements (tungsten, titanium, molybdenum, vanadium) in 14% chromium, 10% manganese steel on rupture strength and tensile properties at room and elevated temperatures. Performance in service. 6 figures.—HB. 16622

6.2.5, 6.3.15, 6.3.20, 8.4.5

Examination of Corrosion Specimens from Slurry Blanket Mockup Run SM-4. S. A. Reed and E. L. Compere. Oak Ridge National Lab. U. S. Atomic Energy Commission Pubn., CF-58-6-59, June 9, 1958, 13 pp. Available from Office of Technical Service, Washington, D. C.

Generally low attack rates were displayed by specimens of type 347 stainless steel, titanium-75A, and Zircaloy-2 which were exposed for 2396 hr in slurry blanket mock-up Run SM-4. The leading coupon of the array, of type 347 stainless steel, suffered more severe attack due to erosive action by the slurry. Coupons of SA-212-B carbon steel were consumed during the run. No stress corrosion cracking was noted on samples, placed in the pressurizer, of types 347 and AM 350 stainless steels, titanium-6Al4V alloy or Zircaloy-2. (auth).—NSA. 16595

6.2.5, 2.3.7, 4.3.3

Results of Stress Corrosion Tests of Type 347 Stainless Steel in 500 F pH 10-11 Lithium Hydroxide. G. E. Galonian. Knolls Atomic Power Lab. U. S. Atomic Energy Commission Pubn., KAPL-M-GEG-11, May 2, 1958, 29 pp. Available from Office of Technical Services, Washington, D. C.

The results of stress-corrosion tests of T-347 stainless steel in 500 F pH 10-11 lithium hydroxide are described. Stressed-bolt specimens heated to 626, 665 and 695 F to produce boiling were exposed for four weeks to determine the extent of corrosion to be expected. Also, tests were made to evaluate corrosion under conditions of leakage to the atmosphere. In the boiling tests, corrosion scales thicker than expected were found, and metal penetration of about 0.001 in. occurred in some areas of the threads on the bolt specimens. No stress-corrosion cracking was found by microscopic or metallographic examination. No deposits of lithium hydroxide were found. Deposits of calcium carbonate were present. Tests to evaluate leakage to the atmosphere were made with a type 347 stainless steel bar stressed in a Schroeder embrittlement detector. A mixture of calcium carbonate with some lithium compounds was found on the test surface after four weeks. No severe corrosion attack was observed. (auth).—NSA. 16521

6.2.5, 6.3.15, 8.4.5

Corrosion of Stainless Steel and Titanium in Purex 2WW. M. C. Fraser. General Electric Co., Hanford Atomic Products Operation. U. S. Atomic Energy Commission Pubn., HW-43986, March 24, 1958, 18 pp. Available from Office of Technical Services, Washington, D. C.

Heat-transfer corrosion rate data are presented which compare the resistance of three types of stainless steel and one type of titanium to corrosion by boiling synthetic Purex 2WW. Specific corrosion parameters investigated were time and temperature. Laboratory and mock-up data had the same order of magnitude. Pilot plant data are presented which illustrate some aspects of the nature of corrosion on heat-transfer surfaces as affected by material composition and method of fabrication. Types 304-L, 347 and 312 stainless steel were tested in the annealed, sensitized, or as-welded condition, and type A-70 titanium was tested in the annealed condi-

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tion. Their corrosion rates were linear with time after an induction period, and, with two exceptions, were linear with temperature. At 1,000 hrs. of exposure and a metal temperature of 114 C, sensitized type 347 had a corrosion rate of about 57 mils/yr, sensitized 304-L—about 39 mils/yr, sensitized 312—about 33 mils/yr and annealed A-70 titanium—about three mils/yr. (auth)—NSA.

16491

6.2.5, 3.7.4, 3.7.3

Sigma Phase in Austenitic Steel E1448. E. I. Uryupina and A. F. Likina. Metalloved. I Obrabotka Metallov, No. 6, 37-41 (1958) June. Translation Available from Henry Brucher, Technical Translations, P. O. Box 157, Altadena, California.

Study of mechanical properties of steel E1448 (0.10% carbon, 1.09 manganese, 0.73 silicon, 16.58 chromium, 11.9 nickel, 0.62 titanium, 1.75 molybdenum) after quenching from 1200 C and 10 to 6000 hours of ageing at 575 or 800 C and effect of presence of sigma phase on them. Electrolytic procedure for isolating sigma; chromium, iron and molybdenum contents in residue as function of ageing time; iron content of residue as index of quantity of sigma present. Ageing temperatures and times required for sigma to form (directly from austenite). Results of microexamination. Presence of sigma as affecting impact strength and reduction of area at room and elevated temperatures; tensile properties in variously heat-treated states. Results of rupture tests. 4 figures, 1 table.—HB.

16161

6.2.5, 8.8.5

Cast Age-Hardenable Austenitic Steels. E. A. Lange, N. C. Lowells and A. Bukowski. U. S. Naval Research

Lab., May, 1958, 16 pp. Available from Office of Technical Services, U. S. Dept. of Commerce, Washington 25, D. C. (Order PB 131733).

Chromium-nickel-phosphorus, chromium-manganese and chromium-nickel-manganese-vanadium types of age-hardenable, austenitic steels which have high strength characteristics in wrought forms were investigated for use as high-strength, nonmagnetic steels for castings. A chromium-nickel-phosphorus austenitic steel with 0.3% carbon and 0.25% phosphorus developed yield strengths at the 100,000 psi level. The chromium-manganese type of austenitic steels containing phosphorus or vanadium were age hardenable, but castings of these alloys were brittle when they were heat treated to high strength levels. Modifying the wrought chromium-nickel-manganese-vanadium composition resulted in an alloy with good ductility and yield strengths at the 100,000 psi level. A fourth type of age-hardenable, austenitic steel, manganese-vanadium, containing a minimum of alloying elements was developed. The hardness and tensile properties of four compositions which were cast and heat treated to yield strengths at the 100,000 psi level are reported.—OTS.

16159

6.2.5

Medium and Low Tungsten High-Speed Steels Alloyed with Cobalt. M. S. Chaadaeva. Metalloved. I Obrabotka Metallov, No. 3, 48-53 (1958) March. Translation available from Henry Brucher, Technical Translations, P. O. Box 157, Altadena, California.

Development of new high-speed steels giving a superior performance in machining operations entailing impact-like

stresses. Compositions of experimental HS steels studied. Conditions of heat treating. Effect of number of temperings on amount of retained austenite and on hardness. Data on transverse bending strength; effect of subzero treating. Results obtained with cyaniding and sulfidizing of a 12% tungsten, 5 chromium, 5 vanadium, 5 cobalt, 1 molybdenum steel. Data on composition of carbide phase; and on the life of cutting tools of the various steels. Evaluation of results: Life of new steels is two to three times that of the conventional 18-4-1 type. 9 figures, 6 tables.—HB.

16463

6.3 Non-ferrous Metals and Alloys—Heavy

6.3.19, 4.3.2, 3.4.3

Corrosion Studies. Pt. XIII. The Course of the Corrosion of Zinc in Chloride Solutions and the Resulting Corrosion Product. I. Sekerka and K. Smrček. (In German.) Coll. Czechoslov. Chem. Commun., 22, No. 3, 712-719 (1957) June.

Zinc specimens (98.5% zinc), 1.5 mm thick and 36 cm² surface area were immersed in distilled water and potassium chloride solutions (10⁻⁴N, 10⁻³N, 10⁻²N, 10⁻¹N and N) for 2 hours, after which pH and concentration of Zn²⁺, Cl⁻ and oxygen were determined. The specimen was immersed in fresh solutions for a further 2 hours and the cycle repeated for a total of 24 hours. Parallel tests were made in which, after 2-hours immersion, the solid corrosion product was dissolved in ammonium hydroxide and analyzed. The results are tabulated and show maximum corrosion in 10⁻²N-hydrochloric acid. The corrosion product consisted of zinc hydroxide with

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increasing zinc chloride as the potassium chloride increased, to a limiting compound $2Zn(OH)_2 \cdot ZnCl_2$. The influence of the corrosion product on the course of the corrosion is discussed. 17 references.—MA. 16318

6.3.6, 4.3.3

Brass Corrosion in Alkaline Medium. (In Russian.) L. S. Zhuravlev and D. Ia. Kagan. *Elek. Stantsii*, 28, No. 12, 26-28 (1957) Dec.

Effect of water alkalinity on corrosion of brass; establishment of conditions for corrosion prevention in heat exchangers; corrosion velocity in various media; dependence of brass corrosion on sodium hydroxide concentration in the presence and absence of oxygen; rela-

tionship between copper and zinc in the corrosion products.—BTR. 16406

6.3.5, 3.5.9

Development of Niobium-Base Alloys. R. T. Begley. Westinghouse Electric Corp. for U. S. Wright Air Development Center, U. S. Air Force, May, 1958, 113 pp. Available from Office of Technical Services, U. S. Department of Commerce, Washington 25, D. C. (Order PB 151004.)

This program was undertaken to evaluate the potentialities of niobium-base alloys for structural applications at temperatures over 1800 F. The work included studies of the creep-rupture properties, flow and fracture characteristics, oxidation behavior and weldability

of commercially pure niobium. Among the results, niobium was found to undergo a ductile-brittle transition in the range —125 to —196C, and its transition temperature range was less affected by the presence of interstitial impurities than many other body-centered cubic metals. Creep-rupture studies at 982 and 1093C showed that the 100-hr rupture strength of commercial niobium in vacuum is significantly greater than that of unalloyed molybdenum. It was suggested that small quantities of gaseous contaminants may be responsible for the high strength of commercial niobium at high temperatures. From weldability studies it was determined that nitrogen is very detrimental to mechanical properties of niobium welds. High-purity niobium, having a hardness of less than 60 VPN, was produced by cage-zone refining techniques.—OTS. 16441

6.3.5

Development of an Oxidation Resistant Columbium Alloy. Status Report No. 17 for the Period: April 16, 1958-August 15, 1958. Rudolph Speiser. Ohio State University, Research Foundation, Columbus, August 18, 1958, 24 pp. U. S. Atomic Energy Commission Pubn., RF Project No. 467, Contract N6 onr-225 (28) (NR039 005).

A preliminary screening of niobium-titanium-zirconium alloys indicated a 50 at.% zirconium-45 at.% niobium-5 at.% titanium alloy to possess the best combination of low oxidation rate and non-porous adherent oxide film. Further tests of this alloy at 900 C, 1000 C and 1100 C showed a rapid initial weight gain which drops after 3 hours. Simultaneous internal and external oxidation occurs resulting in a pearlitic dispersion of oxide phases in a solid solution matrix. The composition, structure and hardness of the oxidized alloy were determined.—NSA. 16660

6.3.5, 3.5.9, 3.2.3

Protection of Niobium Against Oxidation at Elevated Temperatures. Status Report for the period July 16, 1957-March 15, 1958. J. W. Spreynak and Rudolph Speiser. Ohio State University, Research Foundation, Columbus, March 28, 1958, 23 pp. U. S. Atomic Energy Commission Pubn., RF Project 467, Rept. 16, Contract N6 onr-225 (28).

The oxidation behavior of niobium-zirconium alloys from 800 to 1000 C and of niobium-titanium-chromium alloys from 800 to 900 C was observed along with some physical properties of niobates. Oxide coatings on various alloys in the binary and ternary systems were studied in elevated temperature oxidation experiments, and the spallation characteristics of cooled samples described.—NSA. 16659

6.3.5, 3.7.4

Properties of Niobium-Silicon Alloys. (In Russian.) G. V. Samsonov, V. S. Neshpor and V. A. Ermakova. Inst. of Metal Ceramics and Special Alloys, Academy of Sciences, Ukrainian SSR. Zhur. Neorg. Khim., 3, 868-878 (1958) April.

Metallographic and X-ray diffraction investigations of niobium-silicon systems with 0 to 100 at.% silicon showed the existence of three intermediate complexes: the Nb_3Si with hexagonal lattice with constants $a = 3.59$ and $c = 4.46$ Å, the Nb_5Si_3 in three modifications α and β ($a = 6.56$, $c = 11.86$ Å, and $a = 10.00$, $c = 5.07$ Å, respectively) and hexagonal γ modification with constants $a = 7.52$ and $c = 5.24$ Å; and the hexagonal sili-



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cides NbSi_2 with lattice constants $a=4.78$ and $c=6.56$ Å. The melting points and the electric conductivity of the alloys were determined, and hypothetical phase diagrams were plotted on the basis of the data. The corrosion resistance was tested at 1000 C in the air; the systems did not exhibit strong corrosion resistance.—NSA. 16665

6.3.6, 8.8.5

The Chemical Treatment of Copper Alloys. R. W. Ruddle. Foundry Services, Inc. Trans. Am. Foundrymen's Soc., 66, 271-277 (1958); Modern Castings, 33, No. 6, 75-81 (1958) June.

Reviews some of standard chemical methods of treating molten copper alloys and presents some indication of underlying theory behind these treatments. Melting fluxes, degassing procedures and grain refining are dealt with. In considering gas removal, treatments to remove hydrogen and to pre-

vent compound gas formation (steam, sulfur dioxide and carbon monoxide) are discussed separately. Graphs present data for various bronzes, brasses and gunmetals.—INCO. 16604

6.3.8, 4.3.2, 3.7.2

On the Corrosion Resistance of Lead and Lead Alloys (1st. rept.). On the Corrosion Resistance of Lead and Lead Antimony Alloys. F. Hiram and N. Watanabe. J. Japan Inst. Metals, 22, No. 4, 161-165 (1958) April.

The corrosion rates of lead and lead-antimony alloys in sulfuric acid, sulfuric acid + ammonium sulfate solution, sulfuric acid-nitric acid mixed acid, hydrochloric acid, phosphoric acid and phosphoric acid-sulfuric acid mixed acid, have been studied. (1) Below 100 C, there is no difference between lead antimony alloys in resistivity against sulfuric acid. At boiling point, lead and lead-antimony alloys were corroded by

concentrated sulfuric acid over 60 and 70% respectively. (2) Lead-antimony alloys containing more than 2% antimony showed high resistance to sulfuric acid + ammonium sulfate solution while pure lead corroded severely. (3) Lead showed sufficient resistance in sulfuric acid-nitric acid mixed acid at 100 C; lead-antimony alloys are inferior to pure lead in this respect. (4) In hydrochloric acid of concentration below 5%, lead and lead-antimony alloys are corrosion-resistant at 80 C. (5) At 100 C, lead and lead-antimony alloys were corroded by concentrated phosphoric acid over 60%, but showed corrosion resistance in phosphoric acid-sulfuric acid mixed acid.—JSPS. 16502

6.3.10

Nickel Alloys for Controlled Thermal Expansion. E. M. Wise. Inco. Product Eng., 28, No. 17, 68-71 (1957) October 28.

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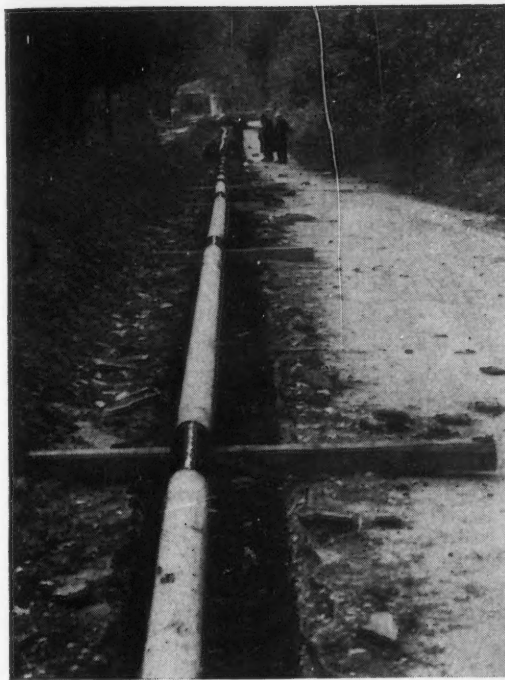
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Review shows that nickel alloys provide potent design tool by combining several levels of corrosion resistance with wide range of thermal expansion characteristics. Considered are: low-expansion alloys (nickel-iron compositions such as Invar, nickel-cobalt-iron alloy Kovar, cobalt-chromium-iron alloys and 400 series stainless steels); high-expansion alloys (Inconel, Monel, nickel-copper, nickel-chromium and 200 and 300 series stainless steels); nickel cast irons (Ni-Resists); bimetals; and glass-metal seals. Table lists average thermal expansion coefficients at 300 F and 650 F. Graphs show inflection temperature and Curie temperatures for nickel-iron alloys and comparison of mean coefficients of nickel cast irons. Applications shown are 1 nickel cast iron-lined alumi-

num brake drum, and high-nickel cast iron insert in aluminum piston.—INCO. 16411

6.3.11, 8.4.5

Silver-Indium-Cadmium Could Replace Hafnium for PWR Control Rods. I. Cohen, E. F. Losco and J. D. Eichenberg. Westinghouse Electric Corp. Nuclear, 16, No. 8, 122-127 (1958) August.

The results of tests made on a silver-15 wt % indium-5 wt % cadmium alloy indicate that it could replace hafnium in control rods of future pressurized-water reactor cores. The effectiveness of the alloy for neutron capture is 99% that of hafnium, and the decrease is small over the core life. It has good corrosion resistance. The alloy has sufficient yield strength to resist scrambling loads of PWR control rods. There

is no change in appearance and mechanical strength improves with irradiation. The melting, forging, extrusion and machining are readily performed. The alloy would be preferable to hafnium because of greater availability and lower cost.—NSA. 16783

6.3.11, 4.7

Iridium as a High-Temperature Material. F. D. Richardson. Platinum Metals Review, 2, 83-85 (1958) July.

Iridium crucibles were completely resistant to attack by lead at 1000 to 1500 C. The crucibles lost weight slightly during use and became etched in appearance, whether they held slags or metal. The metal grains so revealed ranged up to 1 mm across. Typical losses of iridium were: 0.0002 g into 18 g lead in 444 hr at 1000 C, 0.0014 g into 20 g slag and 20 g lead in 144 hr at 1000 C. 6 references.—MR. 16605

6.3.13, 3.2.2

Fragility of Tantalum in the Presence of Hydrogen at Ambient Temperatures. (In French.) Auguste Clauss and Hubert Forestier. Compt. rend. 246, 3241-3243 (1958) June 9.

Experiments with tantalum wires from two sources are described. It was found that hydrogen embrittlement manifested itself only during deformation. The decrease in resistance to drawing varied with the rate of application of force. Study of the modulus of torsion showed that hydrogen does not affect the area of elastic deformation.—NSA. 16436

6.3.15, 2.3.9, 3.7.4, 3.7.3

Metallography of Titanium Alloys. H. R. Ogden and F. C. Holden. Battelle Memorial Inst. for Assistant Secretary of Defense for Research and Engineering. May, 1958, 96 pp. Available from Office of Technical Services, U. S. Dept. of Commerce, Washington 25, D. C. (Order PB 121645).

A general description of titanium metallography as it is affected by composition and thermal treatment is given in this report. Specific illustrations and microstructures of commercial alloys as they are affected by fabrication and thermal treatments are also given. An index is included for the various structures illustrated in sections on commercial alloys. A glossary of terms used in describing titanium microstructures is also presented, along with a description of methods of preparing titanium samples for metallographic examination.—OTS. 16598

6.3.15, 3.5.9, 3.8.4

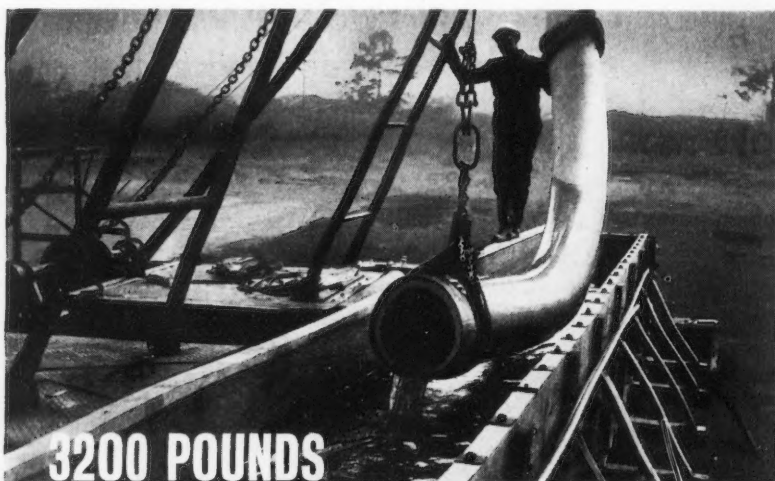
Oxidation of Titanium-Iron Alloys in Air at High Temperatures. (In Japanese.) Morinaga Takuichi, Miura Ishi and Takaai Tetuya. Light Metals, No. 30, 42-52 (1958) May.

Investigated by measuring the weight change due to formation of oxidation layers. Specimens were Kroll titanium, 1% iron, 2% iron, 4% iron and 8% iron alloys heated in air at 600, 700, 800 and 900 C. The parabolic rate law was applicable to the oxidation of Kroll titanium and titanium-iron alloys in the range 600-900 C. Activation energies were calculated.—MR. 16641

6.3.15, 4.3.5, 4.7

Corrosion of Titanium and Titanium-Base Alloys in Liquid and Gaseous Fluorine. G. L. Ericson, W. K. Boyd and P. D. Miller. Battelle Memorial Inst. April 30, 1958, 8 pp. Available from Battelle Memorial Inst., Titanium Metallurgical Lab., Columbus, Ohio.

Unalloyed titanium and four titanium-base alloys were subjected to a simple



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preliminary corrosion evaluation in liquid and gaseous fluorine at temperatures between -320 and $+220$ F. All materials exhibit promising corrosion resistance under the test conditions. More elaborate experiments would be desirable to fully establish the utility of titanium in fluorine service. (auth)—NSA. 16509

6.3.15, 3.6.6, 2.3.5, 4.3.6

The Galvanic Corrosion Properties of Titanium and Titanium Alloys in a Salt-Spray Environment. D. W. Stough, F. W. Fink and R. S. Peoples. Battelle Memorial Institute. October 25, 1957, 15 pp. Available from Titanium Metallurgical Lab., Battelle Memorial Inst., Columbus, Ohio.

It has been found, in galvanic couple tests, that commercially pure titanium and four titanium alloys galvanically accelerate the corrosion of magnesium alloys, aluminum alloys, and mild steel and a straight chromium stainless steel in a salt-spray environment. These same titanium materials showed little or no galvanic effect when coupled to an 18-8 stainless alloy, Monel, or 17-7PH stainless steel in this environment. These experiments also indicated that titanium has slightly less tendency to promote galvanic corrosion than 18-8 stainless steels when compared under salt-spray conditions. (auth)—NSA. 16278

6.3.15, 1.4

Compilation of Available Information on Titanium-6 Aluminum-4 Vanadium Alloy. R. W. Douglass and F. C. Holden. Battelle Memorial Inst. Feb. 28, 1958, 185 pp. Available from Battelle Memorial Inst., Titanium Metallurgical Lab., Columbus, Ohio.

Available data on fabrication, corrosion, heat treatment, mechanical properties and metallurgy of titanium-6 aluminum-4 vanadium alloy are summarized. 73 references.—NSA. 16482

6.3.15, 8.10.2

Evaluation of Extrudability of Titanium Alloys and Alloy Steel. A. Tesmen and George Birman. Loewy Hydropress Div., Baldwin-Lima-Hamilton Corp., New York, 1957, 119 pp. U. S. Atomic Energy Commission Pubn., Contract DA-30-069-ORD-963.

A total of 160 commercially pure and titanium alloy billets and 61 steel billets were extruded under various conditions on a 4000 ton press using 6.9 in. diameter billets. The extrudability of these metals is reported in terms of extrusion pressure as a function of reduction ratio, extrusion temperature and two lubrication practices. Effects of glass lubrication are compared with those of a practice employing graphite-molybdenite grease. A promising method of extruding titanium without lubrication was developed. (auth)—NSA. 16423

6.3.17, 3.4.6, 3.5.9

Final Report on the Results of Exposure of Tuballoy (Uranium) at Elevated Temperature in Air, Nitrogen, and Neutral Atmospheres. W. E. Lindlief and V. C. F. Holm. National Bureau of Standards. U. S. Atomic Energy Commission Pubn., CT-2733, Feb. 10, 1945 (Declassified Feb. 20, 1957), 17 pp. Available from Office of Technical Services, Washington, D. C.

Uranium oxides UO_2 and U_3O_8 were the products of air oxidation of uranium at temperatures between 100 and 500 C. The oxides formed a protective coating

at temperatures of 200 C or lower. Nitrogen reacts with uranium at temperatures up to 850 C to form the nitride, $UN_{1.4}$. Two physically distinct forms of the nitride which was rapidly oxidized to U_2O_5 on exposure to air at high temperatures were formed. Uranium was annealed in the α and β ranges in purified argon and helium and in high vacuum; no true anneal was observed in γ range.—NSA. 16209

6.3.17, 4.3.5, 5.8.1

The Inhibition of Hydrogen Corrosion of Uranium. A. Krieg and J. M. Napier. R. K. Bennett, editor. Union Carbide Nuclear Co., Y-12 Plant. U. S. Atomic Energy Commission Pubn., Y-1151, Dec. 12, 1957, 37 pp. Available from Office of Technical Services, Washington, D. C.

The results of a series of qualitative uranium-hydrogen corrosion tests are presented. The effects of surface treatment and inhibitive agents on the corrosion reaction are discussed. (auth)—NSA. 16232

6.3.17, 3.7.2, 3.7.3, 4.6.2

Corrosion Evaluation of Binary Uranium Alloys in Water at 100 C. H. A. Saller, H. A. Pray, R. F. Dickerson, W. E. Berry and E. L. Foster, Jr. Battelle Memorial Inst. U. S. Atomic Energy Commission Pubn., BMI-971, Dec. 27, 1954 (Declassified Mar. 1, 1957), 38 pp. Available from Office of Technical Services, Washington, D. C.

The corrosion behavior in boiling water of a number of uranium binary alloys was determined. Additions ranging from 2 to 12 at.% of some 42 elements and effect of heat treatment on

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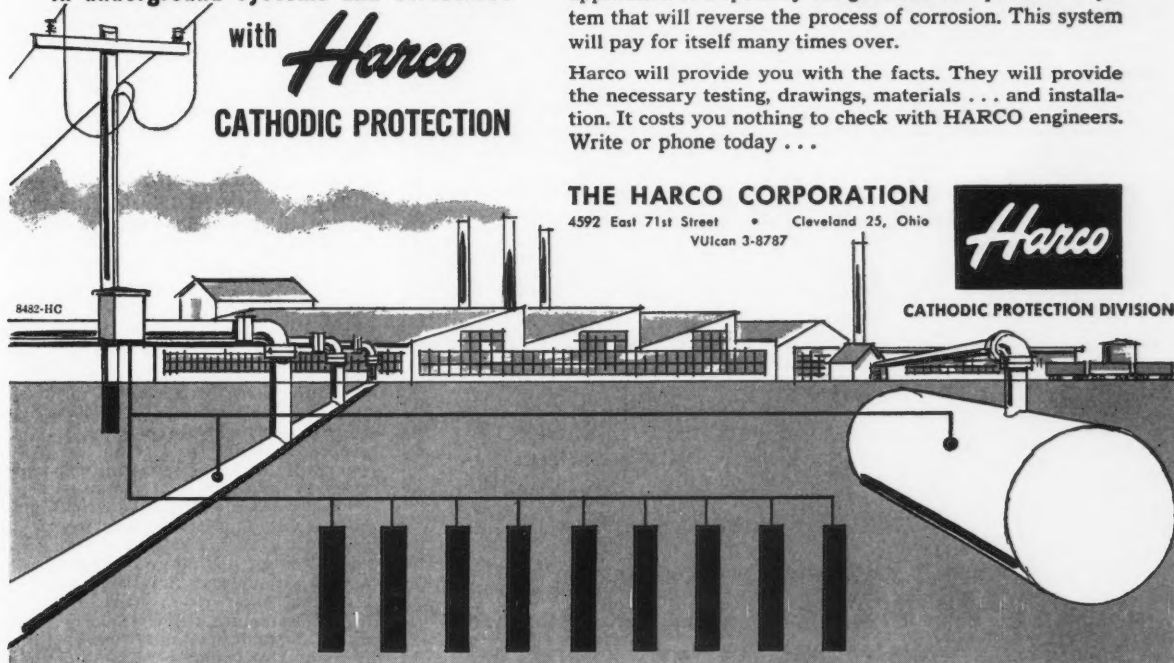
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corrosion resistance were investigated. The alloying was done in an arc furnace designed particularly to expedite melting of a large number of alloys. Melting characteristics, fabrication temperatures and heat-treatment temperatures for each alloy were noted. Vickers hardness numbers of all alloys and data on grain size of the more corrosion resistant alloys were also obtained. (auth)—NSA. 16313

6.3.17

Uranium Alloys: Their Preparation and Fabrication as Zircaloy-2 Clad Fuel Rods. W. B. Haynes. Westinghouse Electric Corp. U. S. Atomic Energy Commission Pubn., WAPD-133, Jan. 31, 1956 (Declassified Feb. 7, 1957), 49 pp. Available from Office of Technical Services, Washington, D. C.

Alloys of uranium-niobium, uranium-niobium-platinum, uranium-molybdenum and uranium-molybdenum-platinum were successfully duplex melted. These alloys were co-extruded in Zircaloy-2, cold drawn and swaged to 0.304-in. diameter.—NSA. 16204

6.3.19, 3.5.8, 3.8.4

Corrosion Studies. Pt. XII. The Influence of a Tensile Stress on the Rate

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of Solution of Metals (Zinc). (In German.) I. Sekerka and O. Vaniček. Coll. Czechoslov. Chem. Commun., 22, No. 3, 705-711 (1957) June.

Zinc (98.5%) wires 1.0 mm in diameter were immersed in normal ammonium chloride plus ammonium hydroxide solutions for periods of 2 hours with and without a tensile stress. The corrosion was indicated by the Zn^{2+} concentration obtained by polarographic analysis of the solution and the results indicate that stress increases the corrosion rate, the effect being partly reversible in the elastic region, but permanent in the plastic range. Experiments at 25, 35 and 45°C are used to show an increase of Arrhenius frequency factor with stress. Experiments with copper wires gave results which were not consistent. The phenomenon is discussed in terms of the generation of new surfaces and breaks in the original surface film. 11 references.—MA. 16317

6.3.20, 3.2.2, 8.4.5, 1.3

Conditions for the Hydriding of Zirconium and Zircaloy. An Interpretive Literature Survey. D. W. Shannon. General Electric Co., Hanford Atomic Products Operation. U. S. Atomic Energy Commission Pubn., HW-55460, April 7, 1958 (Declassified June 19, 1958), 35 pp. Available from Office of Technical Services, Washington, D. C.

A critical review was made of the literature available at Hanford Atomic Products Operation up to March 25, 1958, on the hydriding of zirconium and its alloys. A number of failures of zirconium and Zircaloy tubes and fuel element jacket are described. An hypothesis is set forth on the necessary conditions for hydriding of zirconium and its alloys. This hypothesis is necessarily based on secondary interpretation of experiments largely designed for other purposes, and is subject to modification as more direct experiments are completed. Conclusions drawn from this hypothesis should be considered as probabilities and not as proven facts. One such conclusion is that circumstances exist where the use of Zircaloy jackets and tubes with uranium metal fuel elements could lead to hydrogen embrittlement of the Zircaloy. Uranium metal reacts with water to

form considerable quantities of hydrogen. Under the proper conditions Zircaloy can pick up this hydrogen. Hydrogen concentrations greater than 100 ppm in Zircaloy start to cause hydrogen embrittlement, and very high concentrations can cause loss of all useful mechanical properties. High hydrogen contents can also impair the corrosion resistance of the metal to hot water. Water apparently inhibits the hydriding of Zircaloy and this certainly reduces the possibility of hydriding damage in a water cooled system. However, uranium metal is an excellent dehydrating agent. Thus, dry hydrogen can form and be picked up by the Zircaloy. Once the conditions necessary for hydriding are clearly understood, it should be possible to design the fuel elements and cooling systems so that hydriding probabilities are minimized. (auth) 50 references.—NSA. 16722

6.3.20, 6.3.15, 8.4.5

Recent Advances in the Metallurgy of Zirconium and Titanium Alloys of Special Interest in Reactor Technology. G. M. Adamson, Jr., J. O. Betterton, Jr., J. H. Frye, Jr. and M. L. Picklesimer. Oak Ridge National Lab. U. S. Atomic Energy Commission Pubn., A/CONF. 15/P/1933, 20 pp. Available from Office of Technical Services, Washington, D. C.

Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958.

Zirconium alloys are presently available with better neutron and corrosion properties than any other known reactor material of comparable strength and ductility. Progress has been made towards resolving some of the special problems in these and certain titanium alloys such as fabrication and welding procedures, improved corrosion resistance under heavy particle bombardment, and the delineation of burning hazards. Ultimately, alloy development may permit full utilization of the strong binding forces in these metals, indicated by their high melting point and heat of vaporization, in terms of mechanical strength at higher temperatures. More immediately, alloys which are less sensitive and more resistant to contamination in fabrication are urgently required to reduce the cost of preparing zirconium and titanium materials. (auth)—NSA. 16767

6.3.20, 4.6.2, 3.7.2

The Corrosion of Zircaloy Base Fuel Alloys in High Temperature Water. Stanley Kass. Westinghouse Electric Corp. U. S. Atomic Energy Commission Pubn., WAPD-TM-123, Jan. 1958, 26 pp. Available from Office of Technical Services, Washington, D. C.

The corrosion of Zircaloy-base fuel alloys containing 3 to 8 wt% uranium with and without the addition of boron is characterized by the initial formation of a protective oxide, often followed by a breakdown of the protective film and general spalling. In general, the corrosion resistance decreases with increasing uranium concentration. Zircaloy-2 base fuel alloys containing 5 to 8 wt% uranium exhibit optimum corrosion resistance in the as-fabricated condition. Zircaloy-3 base fuel alloys containing 3 to 8 wt% uranium exhibit best corrosion resistance after quenching from the high-temperature (950°C) field. The addition of small amounts of boron to the Zircaloy-2 or Zircaloy-3 base fuel alloys markedly affects the response of corrosion resistance with prior thermal his-

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tory. Zircaloy-2 uranium-boron alloys exhibit the best corrosion properties after fast cooling from high (950C) temperature, while the low-temperature (550C) treatment results in the most corrosion resistant forms of the Zircaloy-3-uranium-boron alloys. (auth).—NSA. 16575

6.3.20

Progress Report on Metallurgical Studies of Zirconium Alloys. Period Covered November 15, 1957 to February 15, 1958. J. G. Goodwin, J. D. Grozier, L. S. Rubenstein and F. L. Schubert. Westinghouse Electric Corp. U. S. Atomic Energy Commission Pubn., WAPD-NCE-7532, Feb. 15, 1958, 41 pp. Available from Office of Technical Service, Washington, D. C.

The structure, factors affecting the corrosion resistance, and effects of various gas additions on the properties of Zircaloy-2 are described. Brief descriptions of the work done by subcontractors on metallurgical studies of zirconium alloys are included.—NSA. 16520

6.3.20, 4.6.2, 8.4.5

Corrosion Testing of Zircaloy-2 and Zircaloy-3. S. Kass. Westinghouse Electric Corp. U. S. Atomic Energy Commission Pubn., WAPD-TM-97, October, 1957, 32 pp. Available from Office of Technical Services, Washington, D. C.

Zircaloy-2 and Zircaloy-3 have found extensive applications in water cooled and moderated reactors due to their excellent corrosion properties in high-temperature water and steam. These excellent corrosion properties are inherent in the alloys; however, the corrosion behavior in high-temperature water and steam is greatly influenced by sample surface preparation and the manner in which the test is conducted. This report is concerned with the external factors influencing the corrosion behavior of the zirconium alloys.—NSA. 16213

6.3.20, 2.4.3, 8.4.5

Corrosion Test Procedure for the Selection of Crystal Bar Zirconium for Use in Naval Reactor Fuel Elements. (Includes Addendum). V. F. Saitta. Argonne National Lab. U. S. Atomic Energy Commission Pubn., ANL-4450, May 10, 1950 (Declassified Feb. 11, 1957), 12 pp. Available from Office of Technical Services, Washington, D. C.

Reliable test equipment and a reproducible corrosion test procedure have been developed for the inspection and classification of crystal bar zirconium for naval reactor fuel elements. Every bar is examined and classified by a 188-hour high-temperature water corrosion test. This procedure has been instituted by Argonne National Laboratory and is in use by the producers of crystal bar zirconium, the Foote Mineral Co. (auth).—NSA. 16301

6.3.20, 4.7

The Corrosion Resistant Properties of Zirconium. Pt. 2. F. G. Cox. Corrosion Prevention and Control, 5, 46-48 (1958) June.

Summarizes resistance of zirconium to liquid bismuth, bismuth-indium-tin eutectic, bismuth-lead eutectic, bismuth-lead-indium eutectic, gallium, lithium, mercury and sodium. Galvanic corrosion and corrosion by gases. 8 references.—MR. 16449

6.3.20, 8.4.5, 1.3

Tabular Summary of Zircaloy-2 In-Pile Rocking Autoclave Corrosion Data.

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R. J. Davis, Oak Ridge National Lab. U. S. Atomic Energy Commission Pubn., CF-58-6-92, June 18, 1958, 12 pp. Available from Office of Technical Services, Washington, D. C.

A tabular resume of the data from 37 in-pile Zircaloy-2 rocking autoclave corrosion experiments performed since August 1954 is presented. Included are data concerning solutions used on the speci-

mens before and after the reactor exposure, sample treatment prior to irradiation, conditions during exposure, data on oxygen consumption and specimen weight losses, and miscellaneous data.—NSA. 16497

6.3.20

Zirconium Highlights. Westinghouse Electric Corp. U. S. Atomic Energy Commission Pubn., WAPD-ZH-1 and WAPD-ZH-2, Nov., Dec., 1957, 26 pp. and 51 pp. Available from Office of Technical Services, Washington, D. C.

These two reports were issued separately, but are cataloged as a unit.

The following investigations of Zircaloy were made: hydrogen analysis; vacuum vs. atmosphere melting; microstructural appearance of stringers; alpha to alpha-plus-beta transformation temperature; cold-swaging; metallographic etchant; sulfuric acid rinsing after pickling; occurrence of excessive grain growth; anodizing as a technique for determining the origin of stringers; calculation of the diffusion of gaseous contaminants; and effect of removal of surface metal.—NSA. 16401

6.4 Non-ferrous Metals and Alloys—Light

6.4.2, 8.8.5, 2.3.4

Corrosion of Aluminum Die Castings. D. L. Colwell and R. J. Kissling. Paper before Am. Foundrymen's Society, 61st Annual Mtg., Cincinnati, May 6-10, 1957. Trans. AFS, 65, 140-145 (1957); Modern Castings, 32, No. 1, 51-52 (1957) July.

Alternate immersion corrosion tests were made of aluminum die castings in sodium chloride-hydrogen peroxide solution. Tensile and impact bars were immersed for 15 seconds every 15 minutes for periods up to 15 days. Standard mechanical properties of tensile strength, yield strength, elongation and Charpy impact strength were determined after exposure and weight loss was measured on both types of test bars. Silicon alloys have superior corrosion resistance and silicon-magnesium alloy has inferior

corrosion resistance. Silicon-copper alloys are intermediate. Iron beyond 1.3 should be avoided for best corrosion resistance. Zinc up to 2-2½ has no harmful effect on corrosion resistance, as verified by this severe test and by marine atmosphere exposure (Kure Beach). Best resistance to corrosion, as far as regular die-casting alloys are concerned is found with straight silicon alloys. Tables, graphs.—INCO. 16151

6.4.2, 8.1.2

The Corrosion Behavior of Aluminum in the Construction Industry. J. F. Whiting and H. P. Godard. Eng. J., 41, No. 6, 45-54 (1958) June.

The general corrosion behavior of aluminum alloys used in the construction industry and the influence of environmental factors on their performance are discussed. The most common criteria for the evaluation of corrosion resistance are: (1) time to perforation; (2) loss of strength; (3) impairment of appearance and (4) contamination of a product. For roofing, flashings and hot water tanks, resistance to perforation is most important. On the other hand a structural member can perforate without serious loss of strength and, in this case, resistance to mechanical failure is most important. With metal curtain-walls, windows, store fronts and other architectural items, resistance to impairment of appearance is the important and desirable property. Contamination of a product by metal pickup is not usually a problem in the construction industry although it is often critical in chemical and food processing equipment. Behavior of aluminum in natural environments is discussed. Behavior of aluminum in contact with the following classes of building materials is discussed: (1) wood sheathing and building board; (2) concrete masonry and other alkaline building materials; (3) steel, copper and other metals; (4) insulating materials; (5) joint-caulking materials; (6) mastic compounds; (zinc white in oil; white lead, lead wool). The atmospheric performance of anodized, colored and porcelain enamelled aluminum is evaluated. It is stated that aluminum is a preferred metal for many applications, is suitable for most applications and unsuitable only for a few. With good design, correct choice of alloys and normal care during installation, aluminum will give excellent service for a wide range of applications in the construction industry.—ALL. 16716

6.4.2, 4.6.2, 3.7.2

Effect of Alloy Elements on the Corrosion of Aluminum in Water at High Temperatures. (In Norwegian.) K. Videm. Teknisk Ukeblad, 105, No. 18, 409-414 (1958) May 1.

Very pure aluminum corrodes most rapidly in water at temperatures above 130 C, the metal being attacked at grain boundaries. By addition of an alloy element, which produces phases cathodic with respect to aluminum, the attack is retarded. Addition of iron or nickel gives very good results. A range of alloys with varying admixtures of iron and silicon was investigated, the best behaving satisfactorily up to 200 C. At temperatures higher than this alloys with iron and nickel appeared to be superior.—ALL. 16688

6.4.2, 8.4.5

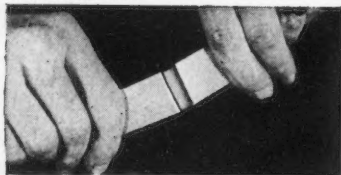
Aluminum and Its Alloys. J. M. Bold. Nuclear Eng., 3, 296-302a (1958) July. The application of aluminum and its alloys as canning materials for reactor

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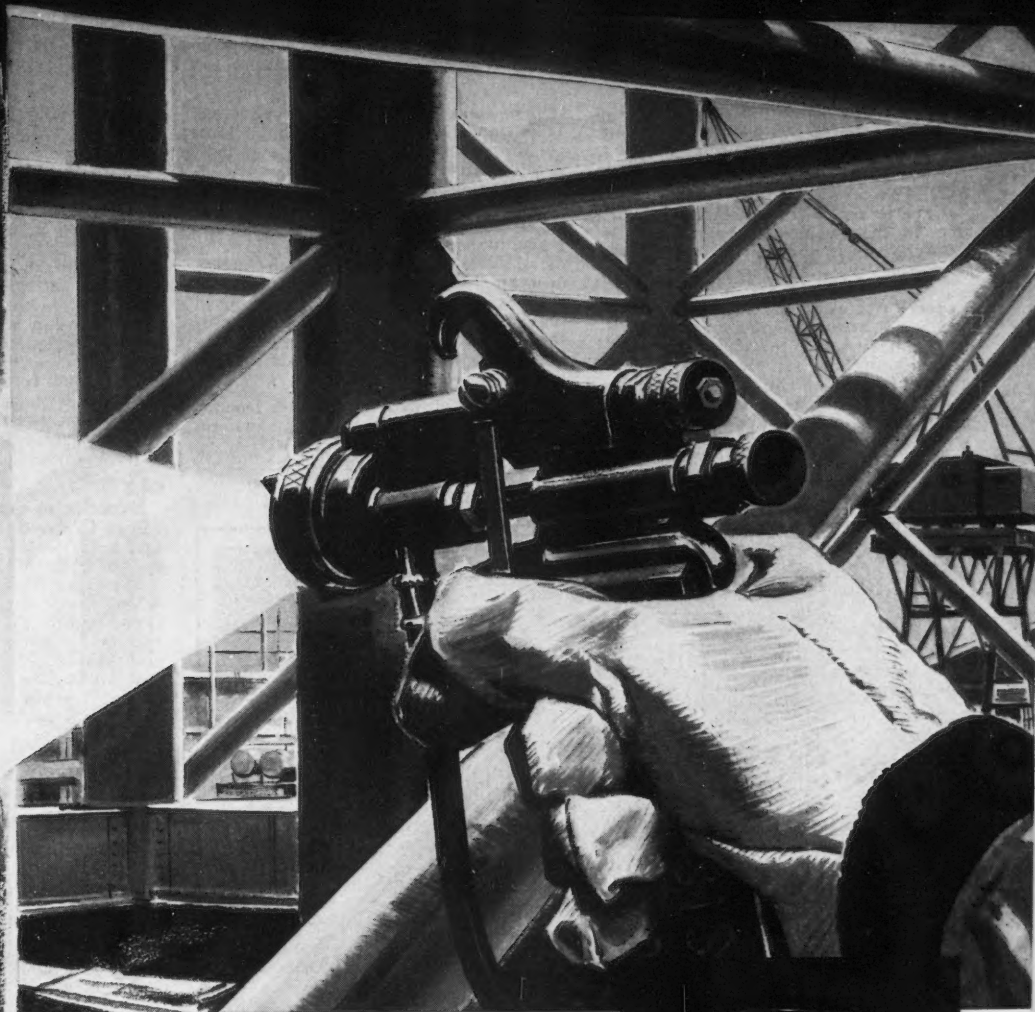
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fuel elements is considered, and the relevant physical, chemical and mechanical properties are discussed.—NSA.

16744

6.4.2, 4.2.7

Experiences with Light Alloy Products in the Tropics. (In German.) M. Rauschert. *Aluminium*, 34, No. 7, 406-409 (1958) July.

The author was given the assignment to carry out weathering tests, in Northern Brazil, with various aluminum products. The investigations extended over a wide range of samples including instruments, such as cameras, ammunition, containers, suitcases, foil, aluminized fabric, etc. Good results were obtained with aluminum-manganese and aluminum-3% magnesium alloys; the conventional methods of protection, such as anodizing, lacquer coating and applica-

tion of light oil proved to be effective. It is important, however, to take local conditions into consideration, viz. temperature changes which may cause condensation in the interior of containers and instruments, cracks in the foil packaging where insects may penetrate, susceptibility of unprotected moving metal parts to corrosion and the action of fungi. There is no general rule for the production of aluminum goods used in the tropics; intimate knowledge of the local conditions is of paramount importance.—ALL.

16599

6.4.2, 4.6.5

Corrosion of 2S Aluminum in Demineralized Water. Progress Report on Problem Assignment No. TX5-12. D. G. Reid and L. P. Bornwasser. Clinton Labs. U. S. Atomic Energy Commission Pubn., MonT-348, July 24, 1947 (De-

classified Feb. 26, 1957), 13 pp. Available from Office of Technical Services, Washington, D. C.

Preliminary corrosion tests have been completed with 2S aluminum exposed to demineralized water at water velocities from 0 to 40 ft/sec, hydrogen peroxide concentrations from 0.05 to 0.0005 molar with the pH maintained at 6, and pH from 3.0 to 6.5 with the peroxide concentration maintained at 0.005 M. The corrosion was studied at temperatures between 80 and 110 C. Corrosion rates as determined by weight losses were generally low. The highest rate obtained in these tests was 10^{-3} in./mo. The pitting was moderate to severe; the maximum pitting rate observed was 0.018 in. in 166 hrs. (auth)—NSA.

16299

6.4.2

Experiences Obtained with Aluminum Alloys Giving Short Turnings. (In German.) H. Greutert. *Aluminium Suisse*, 8, No. 2, 41-47 (1958) March.

Chemical, metallurgical, physical and technological data concerning four aluminum alloys of the Aludur group, developed for the manufacture of products machined on the automatic lathe, are presented. The turnings which are formed on machining these alloys are relatively short and can be removed continuously; this improves the efficiency of the operation. The four alloys discussed are Aludur D 202 (aluminum-copper-lead-bismuth), Aludur D 303 (aluminum-copper-magnesium-lead), Aludur D 404 (aluminum-magnesium-silicon-lead) and Aludur D 505 (aluminum-copper-magnesium-lead). Recommendations are given as to their respective fields of application.—ALL.

16157

6.4.2, 7.7

Structural and Electrical Applications of Aluminum in Europe. Papers before Brit. Aluminum Development Assoc. Symposium, London, May 1957. *Elec. Mfg.*, 61, No. 5, 76-85, 310 (1958) May.

Aluminum, because of its combination of properties, is used in electrical design engineering for conductors, for cable sheathing and for structural equipment in place of steel, cast-iron and zinc base die castings or wrought copper base alloys. It may even supply its own insulating coating when the oxide film is suitably thickened by anodizing. It is of special significance to recall that aluminum has gained a highly important place in the history of the world in less than 70 years. Its industrial use rise is even more remarkable when this life is compared with over 300 years of zinc production and the thousand years of copper and lead. Aluminum has been used in Europe for over a half century and has proved to be an excellent conductor and constructional material, giving no trouble when used correctly. Compositions given of some British aluminum alloy castings used in electrical equipment include nickel content. Photos.—INCO.

16477

6.4.2, 3.8.4

Oxide Coatings on Aluminum Wire. A. A. Defoe. Paper before Wire Assoc., Annual Convention, Chicago, October 1957. *Wire and Wire Products*, 32, No. 11, 1341-1343, 1401-1403 (1957) Nov.

The mechanism of the formation of oxide films on aluminum is reviewed. Initially a film approximately 15-20 Å. thick may be formed in 10-40 minutes, subsequently in humid atmospheres at temperatures greater than 300 C a thickness approximately 10^3 Å. may be



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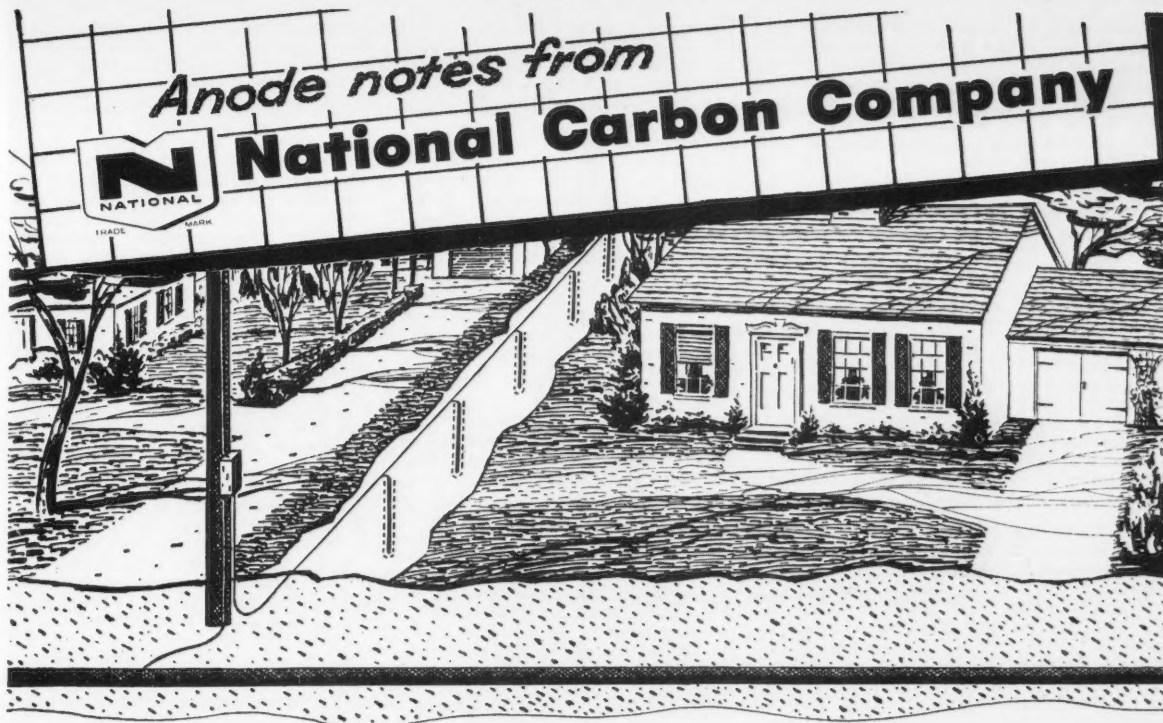
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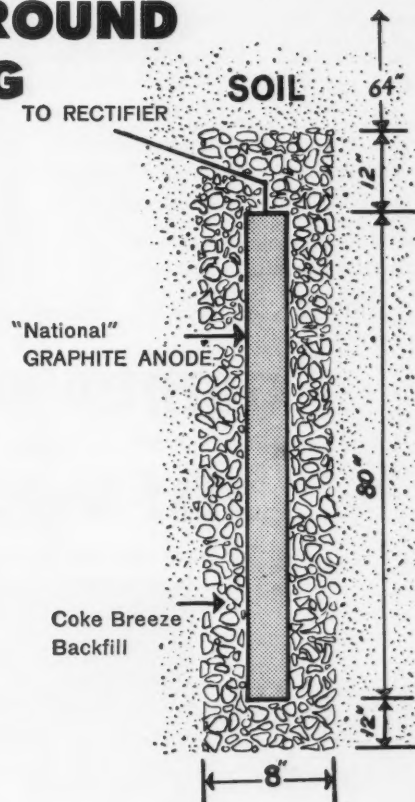
PROTECTION OF UNDERGROUND GAS DISTRIBUTION PIPING

In 1947 an anode comparison test* was sponsored by the TP-3 Subcommittee of the N.A.C.E. In this test, four beds were installed by the Mississippi Power and Light Company with various anode and backfill materials. Each year one quarter of the anodes were removed and inspected.

Based on this test, 2" x 80" graphite anodes in carbonaceous backfill were installed to protect the underground gas distribution lines in March, 1951. These beds have been in operation for more than 8 years with little change in current output.

In bed No. 23, 8 anodes were installed in coke breeze in 15' deep holes spaced on 15' centers. The original bed protected approximately 12,600' of 2" and 5,500' of 3" hot coal tar enamel field coated steel gas lines. Additional pipe has been installed in the intervening years. The bed is still putting out between 8 and 9 amps at 5-7 volts which is sufficient to give a protective potential of .9 volts to a copper — copper sulfate half cell.

*See "Final Report on Four Annual Anode Inspections" by Technical Unit Committee T-2B on Anodes for Impressed Current in CORROSION, Volume 12, 1956, Page 471.



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reached rapidly. Mean d is approximately 3.4 g/cc and melting point approximately 2000 C. The permittivity of the film is quoted as approximately 7.5. Films, 0.005 in. thick, artificially produced by the Alcan process of anodizing aluminum wire, have a dielectric strength of 150-300 V/cm.—MA. 16164

6.4.2, 4.6.5

Corrosion of Aluminum in Deionized Water. Progress Rept. for January 1957-April 1957. R. L. Dillon and R. J. Lob-singer. General Electric Co., Hanford Atomic Products Operation. U. S. Atomic Energy Commission Pubn., HW-49431, March 26, 1957, 9 pp. Available from Office of Technical Services, Washington, D. C.

Static and dynamic tests were made to determine the effects of flow, hydrogen ion concentration, common ion effect and inhibitors on corrosion rates of aluminum alloys. Autoclave studies were designed to determine rates and mechanism of corrosion.—NSA. 16158

6.4.2, 4.3.3

Corrosion of 2-S Aluminum in Potassium Tetraborate Solution. N. R. Miller, Hanford Works. U. S. Atomic Energy Commission Pubn., HW-26928, Jan. 22, 1953 (Declassified Feb. 28, 1957), 7 pp. Available from Office of Technical Services, Washington, D. C.

A laboratory test was conducted to determine the feasibility of using an uninhibited solution of potassium tetraborate in steam condensate as a material for a pile ink facility. The evaluation was made from the standpoints of 2-S aluminum corrosion, the rate and composition of film build-up on aluminum flow surfaces, and the change of solution composition. (auth)—NSA. 16248

6.4.3, 8.10.2, 3.4.9, 3.4.6

The Metallurgy of Beryllium. I. E. Newnham. Research Applied in Industry, 11, No. 5, 185-191 (1958) May.

Discussion of beryllium as to mining and ore dressing, extraction methods, purification and powder metallurgy. Beryllium compares favorably with zirconium so far as resistance to attack

by the common industrial gases is concerned. Unfortunately, the same cannot be said of resistance to aqueous corrosion. Exposure to water containing as little as 0.5 ppm of chloride results in wide spread pitting of beryllium and severe corrosion occurs in a normal saline solution. This type of attack is accelerated by presence of dissolved oxygen, a similar phenomenon being encountered with liquid sodium. Beryllium cannot be safely exposed to liquid sodium containing more than 0.01 oxygen. Health hazards associated with beryllium technology are discussed. Table giving typical impurity content of vacuum-melted beryllium ingot includes nickel content. Photomicrograph, 25 references.—INCO. 16609

6.4.3, 4.6.4, 8.4.5

The Corrosion of Hot-Pressed, Extruded and Vacuum Cast Beryllium in Simulated Cooling Water for the Materials Testing Reactor. James Reed, Oak Ridge National Lab. U. S. Atomic Energy Commission Pubn., ORNL-942, Jan. 9, 1953 (Declassified March 2, 1957), 90 pp. Available from Office of Technical Services, Washington, D. C.

The results of stagnant tests to determine the effects of production method, surface cold work, water quality and metal purity on the corrosion resistance of beryllium to aqueous media are reported. Distilled water, with and without added ionic impurities, containing hydrogen peroxide (0.005 M) at 85 C was the corrosive medium.—NSA. 16306

6.4.4

New Magnesium Alloys. R. J. M. Payne and N. Bailey. Metallurgia, 58, No. 346, 67-68 (1958) Aug.

A series of magnesium alloys has recently been developed by J. Stone and Co. (Charlton) Ltd., for which potential usefulness has been demonstrated in both cast and wrought conditions. Known as the M.S.R. alloys, they contain silver, rare earth metals, zirconium and have higher proof stress figures than existing magnesium alloys. The alloys now being offered for casting

purposes contain 1-3.5% silver, 1-3.5% R. E. metals and 0.4-1% zirconium. Proof stresses exceeding 13½ tons/sq in. have been obtained. The M.S.R. alloys have good casting qualities and are free from hot-tearing or hot-cracking tendencies. They are fully weldable. Pressure tightness is good, as with other magnesium-R.E.-zirconium alloys, and the alloys show a resistance to creep comparable with that of ZRE1 or MCZ at 200 C. The corrosion resistance is not significantly different from, and not inferior to, that of some other magnesium alloys in regular use. The alloys do not appear to be liable to failure by stress corrosion. The use of two-stage heat-treatment processes promote a high degree of uniformity of properties in thin and thick sections of castings and a good correspondence between these and the properties of separately cast test bars.—ALL. 16589

6.4.2, 3.7.2, 3.4.9, 3.5.9, 3.2.2

High Temperature Aqueous Corrosion of Aluminum Alloys. J. E. Draley, C. R. Breden, W. E. Ruther and N. R. Grant. Argonne National Lab. U. S. Atomic Energy Commission Pubn., A/CONF.15/P/114, 18 pp. Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958. Available from Office of Technical Services, Washington, D. C.

It has been shown that exposure of commercial aluminum alloys to water at elevated temperature results in intergranular attack and rapid deterioration. It was also shown that alloying aluminum with nickel and iron largely prevents this type of attack. Since that time a considerable amount of testing has been done at several laboratories, to determine in some detail the corrosion behavior of this type of alloy. The one that has received the most attention is alloy X8001, made by the addition of 1% nickel to American commercially pure metal (known as alloy 1100). Typical significant impurities are 0.5% iron, 0.1 to 0.3% silicon and the order of 0.1% copper. As previously indicated the iron is a desirable constituent of alloy X8001. Efforts were made to develop other alloys with better corrosion resistance. A description is presented of the present state of progress in these objectives. Some of the results have been previously described; some are reported here for the first time. (auth)—NSA. 16781

6.4.2, 8.2.2

The High Abrasion-Resistance of Aluminum and Aluminum Alloys Demonstrated by the Service Records of Coal-Driers. (In French.) Pierre Bandet. Rev. Aluminium, 35, No. 253, 437 (1958) April.

Coal driers used in thermal power generating stations usually consist of a plurality of rotating circular platforms on which the powdered coal is moved by means of scrapers. The conventional material for such platforms is steel. However, the resistance of steel to the combined mechanical and chemical effects of coal is very limited; in fact, the service life of plates 3 mm thick seldom exceeds one year. Experimenting in France with 24S aluminum alloy it was found that after two years of service the aluminum platforms were still in perfect condition. Thus, the industry began to switch over to aluminum alloy drying platforms. As a result of this move about 1,200 aluminum drying units have been ordered so far.—ALL. 16777

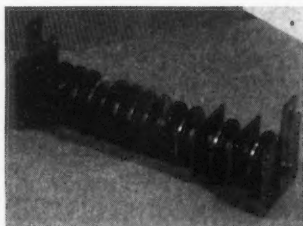
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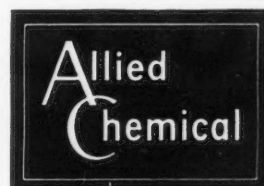
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6.4.2, 5.4.8, 3.6.8, 8.9.5

On the Corrosion of Aluminum Alloy by Ship Bottom Paint and Its Prevention. G. Ito. *Corrosion Engineering (Japan)*, 7, No. 2, 29-33 (1958) March.

The use of aluminum alloy as ship hull plate might lead to the necessity of ship bottom paint which does not corrode aluminum. Anti-fouling paint, containing copper, corrodes aluminum by forming local cell caused by copper precipitate at the defect of paint film. A test using macro corrosion cell of aluminum alloy and copper similar to the above local cell, showed that the corrosion of the aluminum alloy could be inhibited by cathodic polarization and that use of zinc as a protective metal is most suitable for this purpose. It was also found that zinc can protect effectively the corrosion of aluminum alloy by ship bottom paint containing copper as toxic agent.—JSPS. 16567

6.6 Non-Metallic Materials

6.6.4, 3.5.9, 3.4.6

Graphite-Based Materials for High Temperature Applications. Period covered: 12 month period through July 31, 1957. M. Janes. Natural Carbon Company for U. S. Wright Air Development

Center, Technical Report No. 57-602, Dec. 2, 1957, 65 pp. U. S. Atomic Energy Commission Pubn., Contract AF 33-(616)-3537.

Recent aerodynamic developments require a material which will endure under conditions of high heat flux with consequent high surface temperatures and high flow of an oxidizing gas relative to a body of the material. Surface temperature rise may be quite rapid, so that the material must possess resistance to thermal shock. Graphite has outstanding high temperature strength, a high sublimation temperature and excellent thermal shock resistance in comparison to other agglomerate bodies. However, it is quite reactive with oxygen or other oxidizing gas at high temperatures and high relative gas flow rates. The results of a first year of a program to compare the oxidation, with accompanying erosion, of graphite prepared with variation in raw materials or processing, and of combinations of graphite with metals, carbides and compounds such as calcium fluoride are summarized. Three different procedures for testing oxidation-erosion resistance, varying in principle and in severity, were developed and used. Two procedures for evaluating relative thermal shock resistance are described. Graphite

materials prepared by the variations in raw materials and processing so far evaluated, show relatively minor differences in oxidation-erosion rate. Removal of catalytic impurities substantially reduces the rate of oxidation if any portion of the specimen is subjected to relatively low temperature oxidation. Combinations of graphite with various carbides such as those of boron and titanium together with silicon carbide, with the carbide incorporated throughout the body, have shown considerably improved oxidation resistance in an intermediate temperature range (to about 1700 C). It appears possible that further development will extend the stability of these materials to higher temperatures. (auth)—NSA. 16222

6.6.5, 4.3.6

Corrosion of Reinforcing Steel in Concrete in Marine Atmospheres. D. A. Lewis and W. J. Copenhagen. *S. African Industrial Chemist*, 11, 207-219 (1957) October.

Phenomenon of corrosion of reinforcing steel in concrete, causing concrete to spall off, constitutes major problem in South Africa, particularly in coastal areas. It is shown that reaction proceeds by electro-chemical mechanisms whose emf's derive from differences in pH, aeration or other factors but whose severity is influenced mainly by the presence of sodium chloride in the concrete. Accelerated corrosion tests in salt spray cabinet using steel reinforced mortar specimens have indicated that factors of considerable significance are cover to the steel, protective coatings to the mortar, curing, cement content and water:cement ratio of mortar. Effects of these variables on corrosion of steel are discussed. Comprehensive series of both accelerated and natural exposures of reinforced concrete specimens in progress is outlined. Consideration of corrosion prevention covers: location of structure, design, characteristics of the concrete, aggregate and steel, surface coatings, metal treatment, cathodic protection and inhibitors; application of latter two are not recommended at this stage. Graphs show: potential-time curves for steel in cement extracts containing 0.5-10.0 sodium chloride; effect of surface condition of steel on potential; time-potential curves for steel in various neutral salt solutions (up to 20 sodium chloride solution); pH, salt and incidence of rust.—INCO. 16221

6.6.6, 7.1

Refractory Alloys for Aeronautical Gas Turbines and Their Corrosion. (In French.) Marcel Mathieu. *Age Nucléaire*, No. 9, 77-86 (1958) March-April.

Quality requirements of refractory metals used for gas turbines. The modified heat resistant nickel-chromium 80/20 alloys. Cobalt-chromium based alloys. Dry corrosion; scaling. Corrosion of cobalt-base alloys.—BTR. 16552

6.6.6

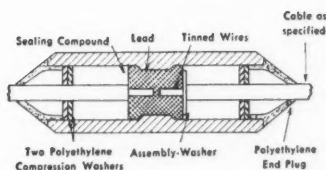
Refractory Metals: Tungsten, Tantalum, Columbium and Rhenium. J. W. Pugh. *General Electric Co. J. Metals*, 10, 335-339 (1958) May.

An analysis of the status and prognosis of the refractory metals tantalum, niobium, tungsten and rhenium with respect to high-temperature applications is reviewed. Tensile properties, creep-rupture strength, oxidation behavior, and oxidation resistant alloys and coatings are discussed.—NSA. 16606

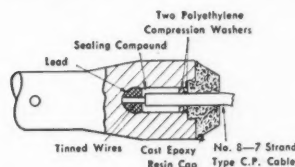
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For more positive cathodic protection of all types of structures used throughout the petroleum industry, insist on DURIRON[®] ANODES. Duriron performs as well as graphite in all the easy services, and far outperforms graphite when the going gets tough in marshlands, at river crossings, or wherever it is impractical to use backfill. Duriron anodes have proven their superiority for the protection of pipelines, drilling rigs, well casings, storage tanks, and similar structures. For real protection, get Duriron.

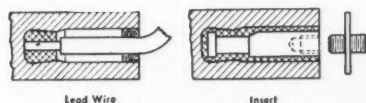
Type G



Types E, J & M



Types B, C & D



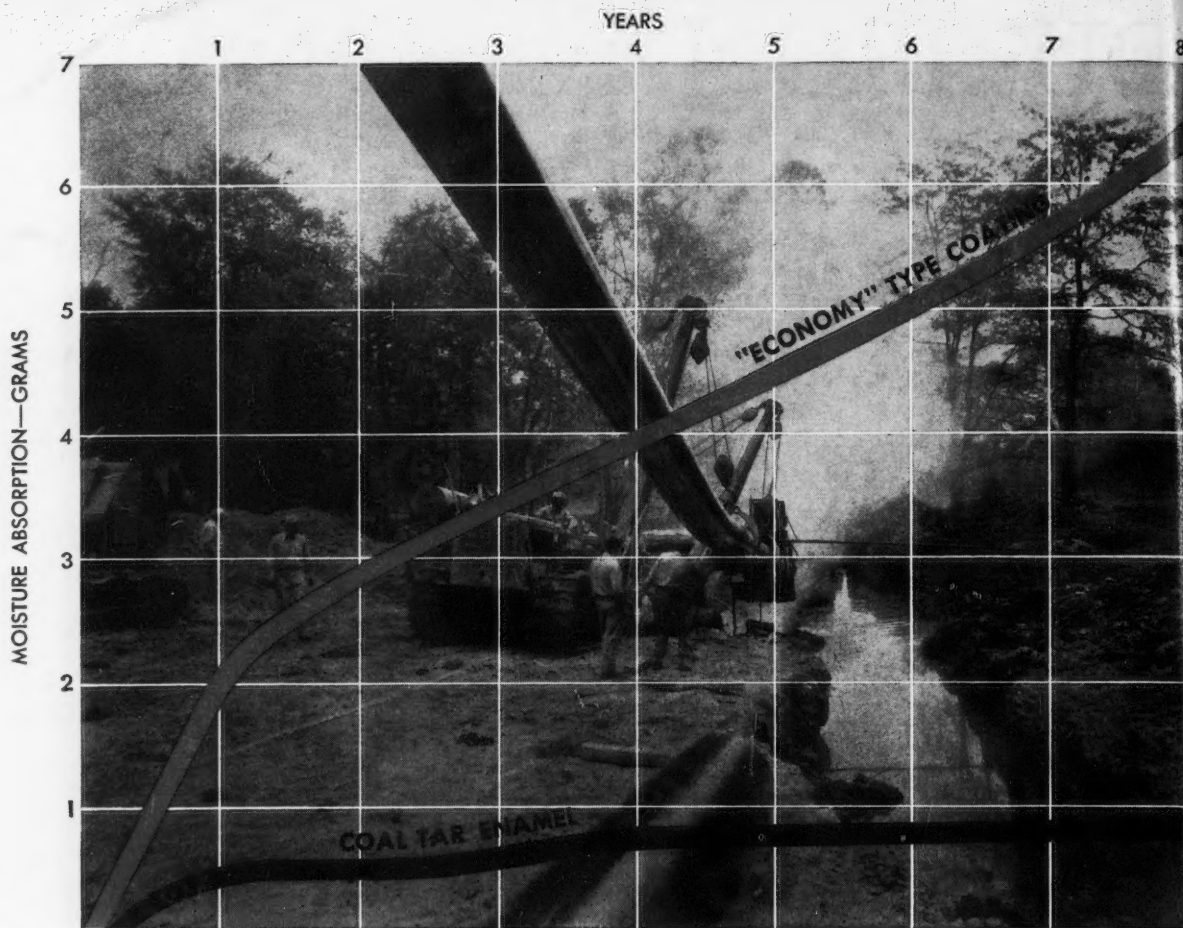
STANDARD ANODE SIZES

Type Anode	Size	Area Sq. Ft.	Weight (lbs.)	General Application
B	1" x 60"	1.4	14	Fresh Water
C*	1 1/2" x 60"	2.0	25	Ground Bed
D	2" x 60"	2.6	46	Ground Bed
E	3" x 60"	4.0	115	Salt Water
G	2" x 9"	0.4	5	Ducts
J	3" x 36"	2.5	80	Salt Water
M	2" x 60"	2.8	57	Salt Water

*Applicable also in fresh water service. Should not be used in ground beds without backfill.

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This Moisture Absorption Comparison Shows Why Most Pipelines are Protected with **COAL TAR ENAMELS!**

THE chart above, prepared by an independent pipeline engineer*, shows why an overwhelming majority of pipeline owners and operators select *coal tar enamels* to protect their underground investment. No other coating material can *begin* to match the ability of coal tar enamel to resist moisture absorption.

Field tests show that an "economy type" coating exhibits a constant moisture absorption increase. The continuously increasing cost of cathodic protection soon eats up any first-cost savings in material. Peeling and deterioration eventually require expensive reconditioning.

So why risk the chance of costly failures with an "economy" coating or other protection methods not *proved by time*? Specify PITT CHEM

- ★ PITT CHEM® Coal Tar Pipeline Enamels
- ★ PITT CHEM "Insul-Mastic"® Gilsonite-Asphalt Coatings
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Coal Tar Coatings and be *sure* of economical, long-lasting protection.

PITT CHEM maintains a full-time staff of field service men to work with your field men in the efficient application of pipeline enamels. *Write us about your protection requirements . . . today!*

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